EXAMINATION OF WATER

FOR SANITARY AND TECHNICAL PURPOSES

LEFFMANN

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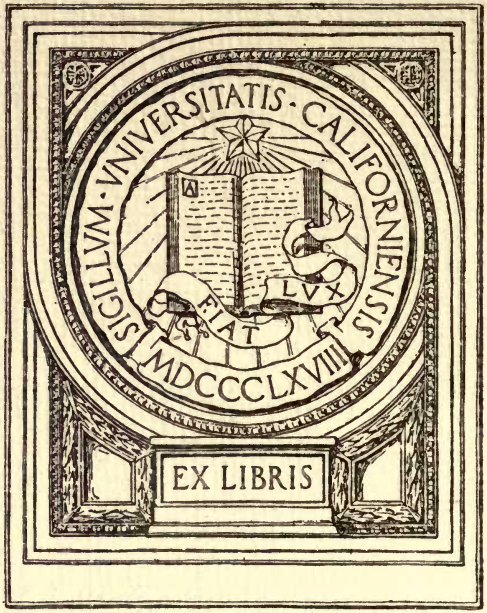
SEVENTH EDITION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EXAMINATION OF WATER

FOR

SANITARY AND TECHNIC PURPOSES.

BY

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PROFESSOR OF CHEMISTRY IN THE WOMAN'S MEDICAL COLLEGE OF PENNSYLVANIA AND IN THE WAGNER FREE INSTITUTE OF SCIENCE; PRESIDENT OF THE ENGINEERS' CLUB OF PHILADELPHIA 1901; VICE-PRESIDENT (BRITISH) SOCIETY OF PUBLIC ANALYSTS 1901–02

SEVENTH EDITION, REVISED AND ENLARGED, WITH ILLUSTRATIONS.

The first two editions of this work were prepared and issued under the joint authorship of Henry Leffmann and William Beam.

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I DEDICATE THIS BOOK TO THE

MEMORY OF

My Mother,

TO WHOSE WISE PRECEPT AND EXAMPLE

IN MY BABYHOOD

I OWE WHATEVER MERIT MY MANHOOD YEARS

MAY SHOW.

313149

—If thou couldst, doctor, cast
The water of my land.

PREFACE.

The favor with which this work was received on the occasion of the issuing of the first edition, and the continuing sale until now the seventh edition is printed, may be taken as evidence that it finds an appropriate place in the library of the chemist.

This edition has been prepared from the point of view of former editions, namely, to furnish the commercial and works-laboratory with a summary of the best processes for ascertaining the sanitary and technical value of a water sample.

Notwithstanding the activity among bacteriologists, the chemical examination of water still holds a prominent and important place, and cannot be neglected in any field of sanitary inquiry.

The present edition has been extensively revised, and the procedures brought into general agreement with those recommended by the American Public Health Association (quoted as APHA). A comparison of the present book with the first edition issued under the joint authorship of Dr. William Beam and myself, will show interesting changes, notably the substitution of proc-

esses devised by American workers for those of foreign origin.

Among the additions are summaries of some of the later researches on corrosion of metals by water, methods of bacteriologic differentiation and details of APHA methods of determining color, turbidity and hardness.

It has seemed premature to adopt the change of expressions for common metric measurement, by substituting liter and mil for c.c., although I favor such change in the interest of accuracy and precision. The error involved is negligible in the class of procedures given in this book. All temperatures are centigrade.

H. L.

PHILADELPHIA.

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NOTE ON WATER-SUPPLY.

The human race must have recognized at an early period the value of an abundant supply of pure and refreshing water. Pastoral communities would be obliged to guide their movements along lines that would keep them within convenient reach of streams and pools for watering cattle; agricultural nations could only establish themselves where water was abundant. Of the two classes, the former would be much less likely to develop soil-pollution, and, accordingly, we find that it is with agricultural and manufacturing centers—*i. e.*, towns and cities—that the serious difficulties with regard to water-supply arise. Drainage areas are easily polluted; in all the civilized countries the streams receive more or less sewage, and are correspondingly offensive to the senses and dangerous to health. The subsoil is better adapted to deal with normal pollution, but its powers are not inexhaustible, and in time wells and springs become unsafe. The usual course of events in this matter is well portrayed by Sextus Julius Frontinus, who was Water Commissioner of Rome from A. D. 97 to 103, and who wrote a comprehensive account of the

water-supply of that city. He says that from the building of the city until its four hundred and forty-first year (312 B. C.), the citizens were supplied with water from the Tiber, or from wells and springs. These springs, as with other nations of antiquity, were often credited with healing powers, either due to some medicinal ingredients or because the locality was supposed to be the abode of a minor deity, whose favor could be secured in various ways. The Roman water-supply became unsatisfactory; aqueducts were built, and these supplied the city for centuries. Two of the original aqueducts are still in use, supplying modern Rome with excellent water. The quantity of water supplied to the ancient city during the height of the development of the aqueduct system—the early part of the second century of the Christian era—is not accurately known, but Clemens Herschel, after careful examination of all data, fixes it at about 50,000,000 gallons a day. Most of the supply was of good quality, but some of the aqueducts delivered river water that was frequently turbid. No proper filtering basins were in use, but special interrupting chambers assisted in getting rid of the coarser suspended matters.

Much less is known of the water-supply of the other great cities of the ancient world. Aristotle, in his "Constitution of Athens," refers to an official

designated "Superintendent of Springs," which shows that municipal hygiene was not wholly overlooked in that city. Jerusalem, during its influential period, was apparently well supplied with water. The remains of underground conduits have been lately discovered, including a tunnel cut through rock to a point distant about 300 meters in a direct line, but an actual length much greater on account of the tortuous course. This tunnel was probably executed in the reign of Hezekiah (eighth century B. C.). An inscription found on the interior wall some years ago, states that the tunnel was bored from each end and the borers met nearly in line. It leads from a spring, now called the Virgin's Spring (considered as identical with the ancient Gihon), to a point near the Pool of Siloam, at the southeast corner of the city.

In common with the general decline of intelligence and morals which affected Europe during the thousand years from the fifth to the fifteenth century, the protection of the water-supply was but little regarded. The aqueducts fell into disuse, and at present are mostly represented by a few scattered ruins studied only by the antiquary and historian.

It is not appropriate to discuss the engineering features of ancient or modern water-supply, but it is worth while to correct a widespread error in

regard to the Roman engineers. It is often said that they did not know that water will rise to its original level, and, therefore, built extensive aqueducts to carry water over valleys. The fact is that they were as fully aware of the principle as modern engineers, but it was usually cheaper for them to build the aqueducts than to use inverted siphons. They did use the latter at times. In the first century of the Christian era, an aqueduct for supplying Lyon (Lugdunum) was provided with a series of seven lead pipes, each about 20 centimeters in diameter, to carry water across a deep valley.

NATURAL HISTORY AND CLASSIFICATION OF WATER.

Pure water is an artificial product. Natural waters always contain foreign matters in solution and suspension, varying from mere traces to very large proportions. The properties, effects, and uses of water are considerably modified by these ingredients, and the object of analysis is to ascertain their character and amount. Since these are largely dependent on the history of the water, a classification based on this will be convenient. We may distinguish four classes of natural waters:

Rain Water.—Water precipitated from the atmosphere under any conditions, and therefore including dew, frost, snow, and hail.

Surface Water.—All collections of water in free contact with the atmosphere, as in streams, seas, lakes, or ponds.

Subsoil or Ground Water.—Water not in free contact with the atmosphere, percolating or flowing through soil or rock at moderate distance below the surface, and derived in large part from the rain or surface water of the district.

Deep or Artesian Water.—Water accumulated

at considerable depth below the surface, from which the subsoil water of the district has been excluded by difficultly permeable strata.

Rain water, when gathered in the open country and in the later period of a prolonged rain or snow, is the purest form of natural water. When collected directly, it contains but little solid matter, this consisting principally of ammonium compounds and particles of organic matter, living and dead, gathered from the atmosphere. In districts near the sea an appreciable amount of chlorids will be present. It is obvious that a prolonged rain will wash out the air, but since storms are usually attended by wind, fresh portions of air are continually flowing in, and thus the water never becomes perfectly pure. Rain water collected in inhabited districts is usually quite impure.

Surface Water.—Rain water in part flows off on the surface, and gains in the proportion of suspended and dissolved matters, the former being found in large amount when the rainfall is profuse. The wearing action of water is dependent on the amount and character of these suspended materials. From the higher levels of a watershed, the streams, more or less in the form of torrents, gather into larger currents, and, reaching lower levels, become slower in movement, and deposit much of the suspended matter.

By admixture of the waters from widely separated districts the character and amount of the dissolved matters are much modified.

It is obviously impossible to establish close standards of composition for surface waters. In the case of rain water, falling on the surface of undisturbed, unpopulated territory, the amount of solids dissolved will be small, and will consist principally of carbonates and sulfates. The water of lakes and rivers is, however, in part derived from springs, which may proceed from great depths, and thus introduce substances not easily soluble in surface water, nor derivable from the soil of the district.

The exposure to light and air which surface water undergoes, results in the absorption of oxygen and loss of carbonic acid, together with the oxidation of the organic matter. The diminution of the rapidity of the current permits the deposition of the suspended matters, and this occurs especially as the river approaches the sea, not only from the retarding influence of the tidal wave, but from the precipitating action of the salt water.

Subsoil Water.—Water that penetrates the soil passes to various depths, according to the porosity and arrangement of the strata. As a rule, it descends until it reaches but slightly pervious formations, upon the level of which it

accumulates. In the upper layer of soil it dissolves mineral and organic ingredients, and becomes impregnated with microörganisms, through the agency of which the organic matter undergoes important transformations. The water, constantly accumulating, gradually flows along the incline of the impervious stratum, or through its fissures, and may either pass downward or emerge in the form of a spring.

Much difference is observed in the composition of subsoil waters, but as a general rule they contain small amounts of mineral substances and organic matter. In populated districts, however, a marked change is produced through admixture with water containing animal and vegetable products in various stages of decomposition. It is especially the organic matter containing nitrogen that is of importance. These are mostly unstable, and decompose, partly by oxidation, partly by splitting up into simpler forms; changes in most cases brought about by microörganisms. The nitrogen is in part converted into ammonium compounds, but a considerable portion suffers further oxidation, and in association with the mineral substances present forms nitrites and nitrates, especially the latter. This is called "nitrification."

Nitrification takes place under the influence of microbes, the habitat of which does not extend

more than a few yards below the surface of the soil. Several microörganisms with active nitrifying powers have been isolated and described. The nitrifying action is often exerted upon the ammonium compounds formed from the organic matter. The presence of some substance capable of neutralizing acids is usually necessary to continuous action. Calcium and magnesium carbonates fulfil this function. Nitrates are the final result of this action; nitrites are present at any given time only in small quantity. Denitrification—that is, the reduction of nitrates and nitrites to ammonium compounds—takes place also under the influence of microbes, and is especially apt to occur when considerable quantities of decomposing organic matter are introduced. Several species of denitrifying bacilli have been described. A partial reduction sometimes occurs, and a notable proportion of nitrites is found, but in the presence of actively decomposing organic matter, such as that in sewage, a complete reduction, even to the liberation of nitrogen, may occur.

Deep Water.—Water which penetrates the fissures of the fundamental rock-formations may pass to great depths, and by following the lines of the lowest and least permeable strata may be transported to points far removed from those at which it was originally collected. The chemical changes thus induced include most of those

which take place at higher points, but the increase of pressure and temperature confers increased solvent power. Carbonic acid will accumulate under conditions favorable to the solution of calcium, magnesium, and iron carbonates, and iron and manganese oxids may be converted into carbonates and then dissolved. Sulfates are reduced to sulfids, and these subsequently, by the action of carbonic acid, yield hydrogen sulfid. Organic matter, living and dead, plays an important part, determining the reduction of ferric compounds to ferrous, and of the sulfates to sulfids, and is itself converted ultimately into ammonium compounds, notable quantities of which are often found in deep waters. Further, it is found that nitrates and nitrites are present only in small amount, except from certain strata rich in organic matter. In some cases the water acquires very high temperature, and dissociation of rocks occurs with solution of considerable amounts of silicic acid, which is ordinarily but sparingly soluble in water.

Masses of water thus accumulated under heat and pressure may find their way to the surface either through natural fissures or be reached by borings.

While no absolute unchangeable line can be drawn between deep and subsoil waters, yet it will in most cases be found that the deep water of a

given district, whether obtained through natural or artificial channels, will be decidedly different in composition from the subsoil or surface water of the same, and that the rocks passed through in such cases will be characterized by one or more strata, difficultly permeable to water, and therefore preventing direct communication. The characteristic differences between surface, subsoil, and deep waters are clearly indicated in the table of analyses given in the appendix.

The fact that mere depth is not the essential difference between the two classes of waters is shown by comparison between the composition of the water (expressed in parts per million) from a well at Barren Hill, near the northern border of Philadelphia County, and a deep well at Locust Point, Baltimore. The former is a dug well, 130 feet deep; the latter is an artesian boring of 128 feet, which in its descent passes through 4 feet of solid rock. The deeper well is evidently supplied by subsoil water. The artesian well, though located about 100 yards from a brackish, sewage-laden estuary, evidently derives no water from it.

	Barren Hill Well.	Locust Point Well.
Total solids, ...	470.00	Less than 100.00
Chlorin,.....	120.00	4.68
Nitrogen as nitrates,	22.00	none

ANALYTIC OPERATIONS.

COLLECTION AND PRELIMINARY EXAMINATION OF SAMPLES.

Great care must be taken in collecting water samples, in order to secure a fair representation of the supply and to avoid introduction of foreign matters. The five-pint green glass-stoppered bottles used for holding acids are suitable for containing the samples. The contents of one such bottle will suffice for most sanitary or technic examinations. Several forms of boxed bottles are now manufactured. Most of these are provided with a hinged lid that can be fastened by a padlock which is sometimes advisable. The green glass-stoppered bottles may be fitted in such an arrangement. Crated demijohns are now made for forwarding water. The larger sizes are well adapted for samples which are to be subjected to elaborate analysis. A boxed bottle called the "New Era demijohn" is also suitable. Stone jugs, casks, or metal vessels should not be employed. The bottles used must be thoroughly rinsed several times with the water to be examined, filled, and the stopper tied down or

fastened by stretching a rubber finger-cot over the stopper and lip. If corks are used, they should be new and thoroughly rinsed. Wax, putty, plaster, or similar material should not be used.

In taking samples from lakes, slow streams, or reservoirs, it is necessary to submerge the bottle so as to avoid collecting any water that has been in immediate contact with the air. In the examination of public water-supplies, the sample should be drawn from a hydrant in direct connection with the main, and not from a cistern, storage-tank, or dead end of a pipe. In the case of pump-wells, a few gallons of water should be pumped out before taking the sample, in order to remove that which has been standing in the pipe.

In all cases care should be taken to fill the vessel with as little agitation with air as possible.

It is important that with each sample a record be made of those surroundings and conditions which might influence the character of the water, particularly in reference to sources of pollution, such as proximity to cesspools, sewers, or manufacturing establishments. The character and condition of the different strata of the locality should be noted if possible.

Determinations of nitrogen existing as ammonium compounds and as organic matter, and of oxygen-consuming power, should be made

upon the sample in the original condition, whether turbid or clear, but all other estimations should be made upon the clear liquid. Turbid waters may be clarified by standing or by filtration; for the latter purpose Schleicher & Schüll's extra heavy No. 598 paper is the best. In many cases the suspended matter can not be entirely removed by filtration, and subsidence must be resorted to. The use of a small quantity of alum, or aluminum hydroxid, as described in the section on the purification of water, will sometimes be applicable as a means of clarifying samples. For the quantitative determination, the sediment from a known volume of the water is collected on a tared filter, dried, and weighed.

FIG. 1.

The water from newly dug wells is generally turbid, and the determinations are best made after filtration; but the results will be unsatisfactory, showing a higher proportion of organic matter than will be found when the supply becomes clear.

Collection of Samples for Bacteriologic Examination.—Bacteriologic examinations are of little

value unless made promptly on samples that have been collected with precautions against contamination. The inoculation of the culture-medium is best done at the source. If this is not possible, glass-stoppered bottles holding about 200 c.c., which have been thoroughly sterilized, with stoppers in place, in a hot-air oven at 100°, must be used for collection. They should be rinsed on the *outside* with the water, dipped below the surface, the stopper withdrawn, and again inserted when the bottle is full. If these are to be transported any distance, they should be packed in ice. For the collection of samples below the surface of the water, the bottle shown in the cut (Fig. 1) is recommended by Abbott. The bottle having been previously thoroughly sterilized is sunk to the proper depth and the stopper is then lifted by a special cord and held until the bottle is full, when, the cord being released, the stopper falls. Before taking out portions for test, the lip and stopper must be thoroughly sterilized by strong alcohol and by careful heating, and, after cooling, washing with sterilized water.

This bottle can also be used for taking samples for chemical analysis except determination of dissolved gases.

Color.—"Apparent" color is that of the water in its raw state, "true" color that of the water

free from suspended matter. Unless otherwise stated, true color is meant. The APHA gives preference to Hazen's method, as follows:

1.246 grams of potassium platinic chlorid (0.5 gram of Pt) and 1 gram of crystallized cobalt chlorid (0.25 gram of Co) are dissolved in water, 100 c.c. of strong hydrochloric acid added, and the solution made up to 1000 c.c. It keeps well, even when exposed to the light. For comparison, 1, 2, 3, etc., of the stock solution are diluted to 50 c.c. in Nessler tubes. These correspond to 0.1, 0.2, 0.3, etc., degrees of the color standard. These also keep for a long time if protected from dust.

Determinations should, of course, be made only on clear samples. The results are expressed in whole numbers, thus:

Color between	1	and	50	to nearest unit
Color between	51	and	100	to nearest 5
Color between	101	and	250	to nearest 10
Color between	251	and	500	to nearest 20

Waters registering above 70 should be diluted and re-examined.

Odor.—This is to be observed at ordinary temperatures (cold odor) and near the boiling point (hot odor).

Cold odor is best observed by shaking a moderate amount of the sample for a few moments in a

stoppered bottle, about half filled, removing the stopper and noting the odor promptly. Hot odor is detected as follows: About 300 c.c. are put into an Erlenmyer flask, the mouth covered with a watch-glass and the liquid heated to just below the boiling point, allowed to cool for five minutes and the odor noted.

The results of odor tests are often valuable as suggestions of certain kinds of contamination, but the data must be used with care, and especially the analyst must not forget that odors may be due to unclean bottles, and very frequently to unclean corks. A common incident of the commercial laboratory is to get a water sample with an odor due to the use of a cork from a wine or whisky bottle or vinegar jug.

Turbidity.—The standard now in use is that adapted by the U. S. Geologic Survey, which is that a turbidity of 100 is that exhibited by a water containing in suspension 100 parts per million of a standard material in such a state of fineness that a bright platinum wire 1 mm. in diameter can just be seen when the center of the wire is 1.2 meters below the surface of the water, the observation being made in the open air in daylight, but not in sunlight and in a vessel so large that the sides do not affect the result.

The material is elutriated fuller's earth, dried, and passed through a 200-mesh sieve. 1 gram of

this in 1000 c.c. of distilled water makes a stock suspension, which should have a turbidity of 1000. This suspension must be tested by diluting a portion of it by nine volumes of clear water and determining if the material has the proper degree of fineness to register 100. If not, it must be corrected by the addition of water or of material as the case may be. From the stock solution standards are prepared by dilution with distilled water. For readings below 20 it is recommended that the standards be kept in bottles similar to those used in collecting samples. For readings from 20 to 100 in 100 c.c. Nessler tubes, about 20 mm. in diameter. Standards must be kept stoppered and be thoroughly shaken before making comparisons. To prevent vegetable growths, a small amount of mercuric chlorid may be added to each.

The standard apparatus for this method is known as U. S. G. S. turbidity rod of 1902. On a glass rod, about 1.5 meters long, is fixed at 25 mm. from one end and at a right angle, a platinum wire 25 mm. long and 1 mm. in diameter. At a distance of 1.2 meters from the center of the cross-section of this wire is fixed a wire ring so that the opening is just above the wire. The point 100 mm. above the center of the wire is marked as 100, and the other proportions are marked as determined empirically by the U. S. G. S., according

to the annexed table, which gives only some of the points. *T* is the degree of turbidity obtained from standards made as indicated above; *V* the distance in mm. above the wire at which the surface of the water touches the rod, when the wire is just at the limit of visibility. Samples that have a turbidity above 500 should be diluted and re-examined, allowance being made for such dilution.

T	*V*	*T*	*V*
7	1095	150	72.0
10	794	200	57.4
15	551	250	49.1
20	426	300	43.2
30	296	400	35.4
40	228	500	30.9
50	187	600	27.7
60	158	1000	20.9
70	138	1500	17.1
80	122	2000	14.8
90	110	3000	12.1
100	100		

Reaction.—Most natural waters are alkaline to the common indicators, but titration often gives a clue to the proportion of carbonates present. Mine waters are often acid, also some deep waters, and occasionally surface waters drawn from areas rich in vegetable matters. Measurements of alkalinity are made regularly in connection with the determination of the several forms of hardness; the standard methods for such purposes are described under that head. It is of some interest to keep a record of alkalinity of

samples in routine laboratory work, which is easily done by titrating 250 c.c. with standard acid using the same indicator for all tests that are to be compared.

ACIDITY.—The most frequent causes of acidity in natural waters are, carbonic acid, iron and aluminum sulfates and sulfuric acid, any or all of which may be present. Distinction may be made by titrating with standard sodium carbonate, using different indicators under special conditions.

Phenolphthalein at room temperature gives the total acidity due to all the above substances. At 100° it gives all except that for carbonic acid.

Methyl orange in the cold gives the acidity due to sulfuric acid.

It will be seen that by these methods the carbonic and sulfuric acidity can be ascertained, and, if the iron is determined by one of the standard methods, that due to aluminum sulfate can be calculated.

TOTAL SOLIDS.

A platinum basin holding 100 c.c. will be found convenient for this determination. This will weigh about 45 grams. It should be kept clean and smooth by frequent burnishing with sand, a little of which should be placed in the palm of the hand, moistened, and the dish gently rubbed

against it. Very fine sea-sand with round, smooth grains is the only kind suitable for this purpose. Coarse river sand, tripoli, or other rough scouring-powders must not be employed. If proper care is taken, the luster of the metal will be retained, and the loss in weight will be trifling. The inner surface can generally be cleaned by treatment with hydrochloric acid, rinsing, and, if necessary, burnishing with sand. Neglect of these precautions will soon lead to serious damage to the dish. A small, smooth slab of iron or marble is convenient to set it on while cooling. When being heated over the naked flame, the dish should rest on a triangle of iron wire, covered with pipe-stems. Dishes of pure nickel are not satisfactory substitutes for those of platinum.

FIG. 2.

Platinum-pointed forceps should be used in handling the dish. The platinum terminals may be kept bright and clean by the use of sand.

The low-temperature burner, used as shown in Fig. 2, will be found a very convenient substitute for the water-bath and hot-air oven. The inlet pipe is very short and soon becomes so hot as to injure the rubber tube. To avoid this it may

be lengthened by means of a piece of $\frac{1}{8}$-inch gas-pipe, or the junction may be wrapped with a rag, the ends of which dip into water. By capillary attraction the rag is kept moist and cool.

The determination of total solids is made by evaporating 100 c.c. of the water in the platinum basin, which has been previously heated almost to redness, allowed to cool for ten minutes, and weighed. The operation is conducted at a moderate heat. When the residue appears dry, the heat may be increased slightly for some minutes. The above method will answer in most cases. In waters of exceptional purity it may be advisable to use larger quantities, such as 250 c.c. When the residue contains deliquescent bodies, the determination will not be accurate, and when appreciable amounts of magnesium and chlorin are present, a decomposition will occur toward the close of the evaporation by which magnesium oxid will be formed and hydrogen chlorid escape.

The irregular decomposition occurring during the evaporation may be largely prevented by adding 0.005 gram of sodium carbonate to each 100 c.c. of the sample taken. This converts magnesium and calcium salts into carbonates. The sodium carbonate is conveniently kept in the form of solution of such strength that 1 c.c. contains 0.001 gram. The weight of the carbonate is, of course, to be deducted from the weight of the

residue. Drown and Hazen have carefully investigated this method and have found it available for a more satisfactory determination of the loss on ignition. For this process they place the platinum basin containing the residue within another similar basin of such size that an air-space of about one-half of an inch is left all around the inner dish, which is supported upon a spiral of platinum that rests on the bottom of the outer dish. Over the inner dish is suspended a disc of platinum foil to reflect the heat. The outer dish is heated to bright redness.

After the weight of the residue is obtained, the dish should be cautiously heated to low redness and the effect noted. Nitrates and nitrites, calcium and magnesium carbonates, are decomposed; ammonium salts are driven off; potassium and sodium chlorids are also driven off if the temperature is high. Organic matter is at first charred, and by continued heating burned off. When the quantity of nitrates is considerable, slight deflagration may be observed, or the production of red fumes of nitrogen dioxid. The organic matter, in decomposing, not infrequently develops odors which indicate its character or source. These are more satisfactorily observed when a rather large quantity, say 250 c.c., is evaporated at a low heat, preferably on a water-bath.

In water of high organic purity, the residue on heating will give no appreciable blackening nor odor, while in forest streams charged with vegetable matter derived from falling leaves, very decided blackening without unpleasant odor will be noticed. The loss of weight after heating can not be taken as a measure of the organic matter, except when present in relatively large amount.

Increase in the price of platinum has led to the use of glass and porcelain dishes. The quartz dishes now available are also suitable. Dishes made of such materials must be carefully cleaned, warmed, cooled (in the desiccator) before each determination. For the accurate determination of the mineral ingredients, evaporation in platinum is indispensable.

CHLORIN.

Reagents:

Standard Silver Nitrate.—Dissolve about 5 grams of pure recrystallized silver nitrate in distilled water, and make the solution up to 1000 c.c. The amount of chlorin to which this is equivalent may be determined as follows: Several grams of pure sodium chlorid are finely powdered and heated over a Bunsen burner for five minutes, not quite to redness. When cold, 0.824 gram are dissolved in water and the solution made up to 500

c.c. 25 c.c. of this should be diluted to 250 c.c. with distilled water and tested as described below, and the amount of silver solution required noted. Each c.c. of the sodium chlorid solution contains 0.001 gram chlorin.

Potassium Chromate.—5 grams of potassium chromate are dissolved in 100 c.c. of distilled water. A solution of silver nitrate is added until a permanent red precipitate is produced, which is separated by filtration.

Aluminum Hydroxid—(For decolorization) 25 grams of ammonium (or potassium) alum are dissolved in 200 c.c. of water, ammonium hydroxid carefully added to slight excess, the precipitate washed until free from chlorids and ammonium compounds and kept in a closed bottle.

Process:

If a preliminary test shows the chlorin to be present in considerable amount, the determination may be made on 100 c.c. of the water without concentration. If, however, there is but little present, 250 c.c. should be evaporated to about one-fifth, with the addition of sodium carbonate, and the determination made on the concentrated liquid after cooling.

The water is placed in a porcelain dish or in a beaker standing on a white surface, a few drops of potassium chromate solution added, and stand-

ard silver nitrate solution run in from a buret until a faint red color of silver chromate remains permanent on stirring. The proportion of chlorin is then calculated from the number of c.c. of silver solution added. Greater accuracy is secured by operating in yellow light. A second determination may be made, using as a comparison the liquid first titrated, the red color having been previously discharged by a few drops of sodium chlorid solution.

The water should always be as nearly neutral as possible before titration. If acid, it may be neutralized by the addition of sodium carbonate. If alkaline hydroxids are present, dilute sulfuric acid should be added until the liquid will just discharge the color of phenolphthalein. If the sample has a distinct color not removable by simple filtration, a volume of aluminum hydroxid (about 3 c.c.) should be added to 500 c.c., the mixture heated to boiling, cooled, filtered and titrated.

NITROGEN IN AMMONIUM COMPOUNDS ("FREE AMMONIA") AND IN ORGANIC MATTER ("ALBUMINOID AMMONIA").

Most natural waters contain ammonium compounds, the exact form being often uncertain, but at the boiling point the carbonates form am-

monium carbonate, which dissociates and distils. From such waters, therefore, the whole of the ammonium compounds can be removed by distillation. If carbonates are deficient, a small pro-amount of sodium carbonate must be added. The nitrogen in the organic matter can be partly converted into ammonium hydroxid by boiling with a strongly alkaline solution of potassium permanganate (alkaline permanganate) and this ammonium hydroxid can also be collected by distillation.

Procedures based on this principle, due to Wanklyn, are extensively used by American and English chemists. The inappropriate terms, introduced by Wanklyn, based on erroneous inferences are still used; the ammonium compounds existing in the water being called "free ammonia" —which they never are—and the ammonium hydroxid obtained from the organic matter being called "albuminoid ammonia"—which it rarely is. It is much better to state the data, respectively as "nitrogen as ammonium" and "nitrogen by permanganate."

Except in waters rich in ammonium compounds, such as sewage and sewage-disposal effluents, in which the ammonium may sometimes be determined directly, the procedures are by distillation and the data obtained by color comparisons with standards of known composition.

Apparatus:

Distilling Apparatus.—Many forms have been devised; Fig. 3 is satisfactory. The flask should be Jena, or similar glass; the condenser is a form devised by Cribb, but modified by Hopkins, by whose name it is generally known. Many

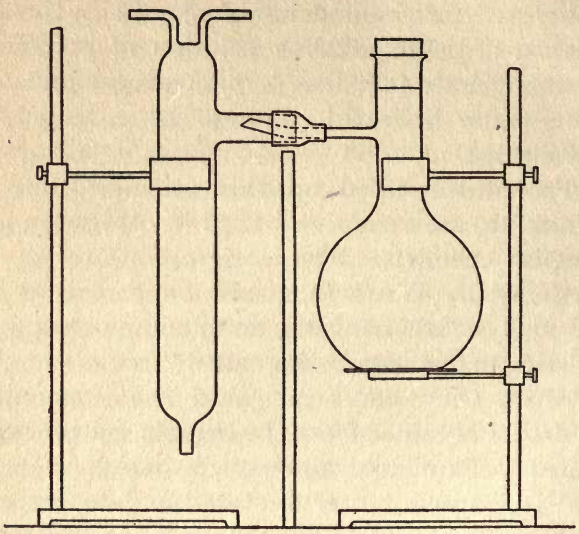

FIG. 3.

chemists prefer block-tin tubes for condensing. The Kjeldahl apparatus in use in the laboratory should not be employed for this work. A stout piece of asbestos should be placed between the flask and condenser, as shown, to avoid radiation of heat.

Nessler Glasses.—Narrow cylinders of colorless glass about 15 mm. in diameter polished on the outside of the base. Dealers now furnish standard forms. In selecting a set, tubes as nearly uniform as possible should be chosen.

Reagents:

Ammonium-free Water.—If the distilled water of the laboratory gives a reaction with Nessler reagent, it should be treated with sodium carbonate, about 1 grain to the liter, and boiled until about one-fourth has been evaporated. Ammonium-free water may be obtained by distilling, in a retort, ordinary water made slightly acid with sulfuric acid.

Messrs. J. B. Weems, C. E. Gray, and E. C. Myers recommend the following method: Sodium dioxid is added to ordinary water in the proportion of 1 gram to 1000 c.c. and the liquid boiled for thirty minutes, or longer if the amount of ammonium compounds is high. It is then cooled and the flask kept closed. Flasks holding several 1000 c.c. are most convenient. If the water is distilled, the distillate may also be used for the preparation of standard nitrate and nitrite solutions.

Standard Ammonium Chlorid.—Dissolve 0.382 gram of pure dry ammonium chlorid in 100 c.c. of ammonium-free water. For use, dilute 1 c.c. of this solution with pure water to 100 c.c. 1 c.c.

of this dilute solution contains 0.00001 gram of nitrogen.

Nessler's Reagent.—(APHA formula). Dissolve 50 grams of potassium iodid in a minimum quantity of water; add saturated solution of mercuric chlorid in small portions with constant stirring until a slight precipitate persists; add 400 c.c. of a clear 50 per cent. solution of potassium hydroxid, stir, dilute to 1000 c.c., allow to settle and decant. The solution should give the full color reaction within five minutes, and should not give a precipitate in two hours with small amounts of ammonium compounds. Nessler and similar reagents should be kept in capped bottles as shown in Fig. 4 in which the pipet may remain when not in use.

FIG. 4.

Alkaline Permanganate.—8 grams of potassium permanganate and 200 grams of potassium hydroxid of good quality are dissolved in 1000 c.c. of distilled water. It is customary to boil this liquid to purify it from ammonium compounds and organic matter producing ammonia, and it is also recommended to make blank experiments with the portions taken for use, but the procedure of twin distillation, herewith described renders these manipulations unnecessary.

Pumice.—Fragments of pumice are kept in a wide-mouth stoppered bottle with water. They

soon become water-logged. If it is desired to prepare pumice for immediate use, fragments should be heated to near redness and plunged into water.

Process:

The distillation apparatus is set up in pairs; each flask is rinsed and charged with 200 c.c. of pure water. To one is added a pinch of sodium carbonate: to the other 50 c.c. of the alkaline permanganate. A few fragments of pumice are put in each to prevent bumping. The liquids are distilled at such a rate that 50 c.c. pass over from each flask in about ten minutes, and the distillates tested for ammonium compounds, as noted below, and when no more is coming off, which will generally be when the volume is reduced to 100 c.c., 250 c.c. of the sample are added to each flask and the distillation resumed. Long-stemmed funnels are convenient for introducing liquids. The distillates are collected in Nessler tubes in quantities of 50 c.c. each and tested as described below.

The flask containing sodium carbonate furnishes only the ammonium existing in the sample (so-called "free ammonia"); the flask containing the alkaline permanganate furnishes also the preexisting ammonium and, in addition, the ammonium formed from the organic matter. Therefore, the figure obtained from distillates from the sodium carbonate should be deducted from that obtained from the other flask; the remainder will

be the ammonium formed from the organic matter (so-called "albuminoid ammonia").

The determination of the amounts of ammonium compounds in the distillates is called "nesslerizing" and is conducted as follows:

Standards are prepared by charging Nessler tubes with the following amounts of the dilute ammonium chlorid solution (see page 25) 0.1 c.c., 0.2 c.c., 0.5 c.c., 1.0 c.c., 1.5 c.c., 2.0 c.c., and so on according to the probable amount to be measured, not going above 6.0 c.c. Each tube is then filled to the 50 c.c. mark with water free from ammonium compounds. The standard tubes and those containing the distillates are placed so that they can be compared by looking vertically through the liquids and 2 c.c. of Nessler reagent added to each running this in at the top of the liquid and allowing it to diffuse by gravity. At the end of ten minutes the colors in the distillates should be compared with those of the standard tubes. Equality in depth of color indicates equality of ammonium content. Each c.c. of the dilute ammonium chlorid solution contains 0.00001 of nitrogen. Standards and distillates must be compared at the same temperature. Many analysts keep each 50 c.c. of distillate separate for test. With waters containing but little ammonium compounds or organic matter, the distillates from a given flask may be mixed, made up to defi-

nite volume with pure water, an aliquot part tested and calculation made.

The comparison standards must be made up each day as they do not keep. If the color in any distillate is very deep, the liquid may be diluted considerably with a known volume of pure water, 50 c.c. of this compared and calculation made. When the amount of ammonium hydroxid in the distillate is considerable a precipitate may occur. Such a result is useless and a new distillation must be made using either less of the sample or diluting the distillate considerably with pure water.

As small quantities of ammonium compounds and nitrogenous matters are everywhere present, the greatest care should be exercised in order to avoid their introduction in any way during the course of the analysis. All measuring vessels, cylinders, etc., should be thoroughly rinsed before using.

TOTAL ORGANIC NITROGEN

Several processes for the determination of the organic nitrogen in water, based on those in use in ordinary organic analysis, have been devised.

The ease and certainty with which the nitrogen of most organic bodies may be converted into ammonium sulfate by boiling with sulfuric acid, offers a means of determination free from the objections of former methods. The method intro-

duced by Kjeldahl for general organic analysis was first successfully applied to water analysis by Drown and Martin.

In their original process, 500 c.c. was concentrated to about 300 c.c., and the distillate nesslerized for determining the nitrogen existing as ammonium compounds. The organic nitrogen was then determined in the residual water. Owing to the fact that organic matter may be decomposed by moderate heat, there is liability to underestimation of the nitrogen. The following procedure suggested by Leffmann and Beam, eliminates that loss and enables determinations to be made without distillation.

Reagents:

Concentrated Sulfuric Acid.—This should not contain appreciable quantities of nitrogen. It can now be obtained of high purity.

Sodium Hydroxid Solution.—The white granulated material sold for domestic uses (concentrated lye) will answer 100; grams are dissolved in about 1000 c.c. of water, the liquid boiled down to one-half volume and diluted to 1000 c.c. by addition of water that gives no color with Nessler reagent.

Sodium Carbonate Solution.—200 grams of good sodium carbonate are dissolved in about 1000 c.c. of water and the solution boiled briskly until several hundred c.c. are driven off.

All reagents must be kept in well-stoppered bottles, protected from dust and fumes of ammonium compounds.

Process:

Nitrogen as Ammonium.—200 c.c. of the water are placed in a stoppered bottle, 2 c.c. each of the solutions of sodium carbonate and sodium hydroxid added, the stopper inserted, the solutions mixed and allowed to stand for an hour or two. A filter is prepared by inserting a rather large plug of absorbent cotton in a funnel. This should be washed with ammonium-free water until the filtrate gives no color with Nessler reagent. The clear portion of the sample is drawn off with a pipet and run through the filter, the first portions being rejected, since it is diluted by the water retained in the cotton. The filtration is rapid; when sufficient filtrate is collected, an aliquot part is nesslerized and the calculation for the whole volume made.

Total Organic and Ammoniacal Nitrogen.—500 c.c. of the sample are mixed with 10 c.c. of the sulfuric acid, boiled until the water is all evaporated, and the remaining liquid colorless or pale yellow, a small amount of potassium permanganate added and the liquid again boiled until colorless, which will require but little time. If the permanganate produces a purple solution at first, the original heating was not sufficient, and

the mass should be heated for some extra time after the color produced by the permanganate is discharged. After completion of the heating the liquid is cooled to room temperature, 50 c.c. of ammonium-free water added, then sufficient of the sodium hydroxid solution to render the liquid slightly alkaline—about 150 c.c. will be usually required—and 2 c.c. of sodium carbonate solution to precipitate the calcium and magnesium. The mixture is cooled by immersion of the flask in water, made up to 250 c.c., transferred to a stoppered bottle, allowed to settle—about an hour will be usually required—sufficient of the clear liquid filtered as in the ammonium determination, and an aliquot part nesslerized.

Of course, the ammonium obtained in the first process is deducted from that obtained in the second, making due allowance for the different proportions of the sample used in the two cases; the remainder is the nitrogen existing in organic matter.

Blank experiments should be made to determine the purity of the reagents.

NITROGEN AS NITRATES.

Reagents:

Standard Potassium Nitrate.—0.722 gram of potassium nitrate, previously heated to a tem-

perature just sufficient to fuse it, are dissolved in water, and the solution made up to 1000 c.c. 1 c.c. of this solution will contain 0.0001 gram of nitrogen.

Phenoldisulfonic Acid.—Strong sulfuric acid and pure phenol are mixed in the proportion of 37 grams of the former to 3 grams of the latter, and heated for six hours *in, not upon,* the water-bath. The resulting compound usually solidifies to a white mass on standing, but can be easily liquefied on the water-bath during the evaporation of the samples to be tested.

The above method of preparing the reagent is recommended by the APHA, and is in general use by American chemists. Many English and Continental chemists use a reagent prepared without long heating, which is less satisfactory. Messrs. Chamot, Pratt and Redfield have shown, however, that even the long heating—first suggested by Gill—does not produce wholly satisfactory results. They give the following as an improved formula:

25 grams pure, colorless phenol are dissolved in 150 c.c. of sulfuric acid (sp. gr. 1.84), 75 c.c. of fuming sulfuric acid (containing not less than 15 per cent. of sulfuric anhydrid) added, the mixture stirred well and heated to 100° for two hours. It is used the same as the other reagent.

Process:

A measured volume of the water is evaporated just to dryness in a porcelain basin about 6 centimeters in diameter. 1 c.c. of the phenoldisulfonic acid is added and thoroughly mixed with the residue by means of a glass rod. The liquid is then diluted with about 25 c.c. of water, ammonium hydroxid added in excess, and the solution made up to 50 c.c.

1 c.c. of the standard solution of potassium nitrate is now similarly evaporated in a platinum basin, treated as above, and made up to 50 c.c. The color produced is compared to that given by the water, and one or the other of the solutions is diluted until the tints of the two agree. The comparative volumes of the liquids furnish the necessary data for determining the amount of nitrate.

The results obtained by this method are satisfactory. Care should be taken that the same quantities of phenoldisulfonic acid are used for the water and for the comparison liquid.

With subsoil and other waters probably containing much nitrates, 10 c.c. will be sufficient; but with river and spring waters, 25 c.c. may be used. When the organic matter is sufficient to color the residue, it will be well to purify the water by addition of aluminum hydroxid and filtration, before evaporating.

Chlorin interferes with the accuracy of the test, but Gill finds that when not amounting to more than 20 parts per million it does not impair the practical value of the results. When greater than this, it is best to evaporate in vacuo over sulfuric acid. If the chlorin be more than 70 parts per million, it should be considerably reduced by the addition of silver sulfate which has been ascertained to be free from nitrates. Nitrites do not influence the reaction.

NITROGEN AS NITRITES.

The following is Ilosvay's modification of Griess's test. It has the advantage over the original method, that the color is developed more rapidly, and the solutions are less liable to change.

Reagents:

1–4-*aminobenzenesulfonic Acid Solution* (*Sulfanilic Acid*).—Dissolve 0.5 gram in 150 c.c. of diluted acetic acid (sp. gr. 1.04).

α-aminonaphthalene Acetate Solution.—Boil 0.1 gram of solid α-aminonaphthalene (α-naphthylamin) in 20 c.c. of water, filter the solution through a plug of washed absorbent cotton, and mix the filtrate with 180 c.c. of diluted acetic acid. All water used must be free from nitrites, and all vessels must be rinsed out with such water before tests are applied, since appre-

ciable quantities of nitrites may be taken up from the air.

Standard Sodium Nitrite.—0.275 gram pure silver nitrite is dissolved in pure water, and a dilute solution of pure sodium chlorid added until the precipitate ceases to form. It is then diluted with pure water to 250 c.c. and allowed to stand until clear. For use 10 c.c. of this solution are diluted to 100 c.c. It is to be kept in the dark. 1 c.c.of the dilute solution is equivalent to 0.00001 gram nitrogen.

The silver nitrite is prepared thus: A hot concentrated solution of silver nitrate is added to a concentrated solution of the purest sodium or potassium nitrite available, filtered while hot, and allowed to cool. The silver nitrite will separate in fine, needle-like crystals, which are freed from the mother liquor by filtration by the aid of a filter pump. The crystals are dissolved in the smallest possible quantity of hot water, allowed to cool, and again separated by means of the pump. They are then thoroughly dried in the water-bath, and preserved in a tightly stoppered bottle away from the light. The purity may be tested by heating a weighed quantity to redness in a tared porcelain crucible and noting the weight of the metallic silver. 154 parts should leave a residue of 108 parts silver.

Process:

25 c.c. of the water are placed in a color-comparison cylinder, and 2 c.c. each of the test solutions are dropped in. It is convenient to have a pipet for each solution, and to use it for no other purpose.

1 c.c. of the standard nitrite solution is placed in another clean cylinder, made up with nitrite-free water to 25 c.c. and treated with the reagents as above.

In the presence of nitrites a pink color is produced. At the end of five minutes the two solutions are compared, the colors equalized by diluting the darker, and the calculation made as explained under the determination of nitrates.

OXYGEN-CONSUMING POWER.

All organic materials being more or less easily oxidized, several methods have been suggested for determining the oxygen-consuming powers of waters by treatment with active oxidizing agents. These methods are, however, limited in value. The organic matters in water differ much in character and condition, and their oxidability is subject to much variation, according to the circumstances under which the test is made. Nevertheless, as a high oxygen-consuming power indicates departure from purity, additional evidence may be obtained by ascertaining it.

It must not be overlooked that if a water contains nitrites, ferrous compounds, or sulfur compounds other than sulfates, the proportion of oxygen consumed will be greater than that required for the organic matter. It has been proposed, in order to remove the nitrites before applying the permanganate, to take 500 c.c. of the water, add 10 c.c. of the dilute sulfuric acid, boil for twenty minutes, allow to cool, and then treat with permanganate. Since, however, the amount of nitrites, if appreciable, can be directly determined, it is more satisfactory to deduct from the oxygen consumed the amount required to convert the nitrites present into nitrates, and the remainder will be that required for the other oxidizable ingredients. 14 parts of nitrogen existing as nitrite require 16 parts of oxygen for conversion into nitrate. Similarly, 111.8 parts of iron in a ferrous compound will require 16 parts of oxygen for conversion to the ferric condition.

Oxygen-consuming power may be determined indirectly by the action of the organic matter upon silver compounds. Fleck's method depends upon the reduction produced by boiling the water with alkaline solution of silver thiosulfate and determination of the unreduced silver. A. R. Leeds proposed a method by treating the water with silver nitrate, exposing to light until it

settles perfectly clear, and determining the reduced silver.

These methods are open to the same objections as in the use of permanganate, and do not seem to possess any decided advantage. Qualitative results of some interest may occasionally be obtained by the following method: 2 c.c. of a 1 per cent. solution of silver nitrate, rendered alkaline by ammonium hydroxid, are added to 100 c.c. of the water in a stoppered bottle, which is then placed in full sunlight for two hours. Waters containing but little organic matter will not show at the end of this period any appreciable tint.

The following procedure for direct oxidation is recommended by the APHA, and is probably as satisfactory as any yet proposed.

Reagents:

Dilute Sulfuric Acid.—Add 100 c.c. of sulfuric acid to 300 c.c. of distilled water, and potassium permanganate solution until a slight pink tint persists for several hours.

Standard Potassium Permanganate.—0.4 gram of pure potassium permanganate dissolved in 1000 c.c. of water. 1 c.c. contains 0.001 gram available oxygen.

Standard Oxalate.—0.888 crystallized ammonium oxalate dissolved in 1000 c.c. of water. 1 c.c. of this requires 0.001 gram of oxygen for complete oxidation. The solution should be com-

pared with the permanganate solution and the exact ratio noted.

Process:

100 c.c. of the sample are measured into a flask, 10 c.c. each of the acid and permanganate solution added, the liquids mixed well and the flask immersed in boiling water, the level of which is above the level of the liquid in the flask. After thirty minutes heating, the flask is removed from the bath, 10 c.c. of oxalate solution added and the mixture titrated with permanganate. A blank experiment with distilled water must be made and the amount of permanganate deducted from that used in the test; the remainder will be the permanganate used by the organic matter.

The ratio of the oxalate and permanganate will usually not be the same at the boiling heat as in the cold, hence the necessity for frequent verifications of this ratio under conditions as near as possible to those of the actual test.

PHOSPHATES.

The following method is recommended by A. G. Woodman:

Reagents:

Ammonium Molybdate Solution.—50 grams in 1000 c.c. distilled water.

Nitric acid, sp. gr. 107.

Sodium Phosphate Solution.—0.5324 gram crystallized disodium hydrogen phosphate, 100 c.c. of the above nitric acid, and distilled water sufficient to make 1000 c.c. This is equivalent to 0.0001 P_2O_5 in 1 c.c.

Process:

50 c.c. of the sample and 3 c.c. of nitric acid are evaporated to dryness in a porcelain dish on the water-bath, the residue heated for two hours at 100°, and treated with 50 c.c. of cold distilled water added in several portions which are mixed in a comparison tube. It is usually unnecessary to filter. 4 c.c. of ammonium molybdate solution and 2 c.c. of nitric acid are added, the contents mixed, and after three minutes the color compared with that given by different quantities of the standard phosphate solution which have been made up to 50 c.c. and the reagents added in the same amount as above.

Blank tests must be made to determine the purity of the materials used. Distilled water kept for some time in glass vessels may acquire appreciable amounts of substances giving color with the reagents.

Acid ammonium molybdate solution suitable for the gravimetric determination of phosphates may be prepared as follows:

Weigh into a beaker 10 grams of pure molybdenum teroxid, mix well with 40 c.c. cold dis-

tilled water, and add 8 c.c. strong ammonium hydroxid (sp. gr. 0.900). When completely dissolved, filter and pour slowly, with constant stirring, into a mixture of 40 c.c. of nitric acid (sp. gr. 1.42) and 60 c.c. of water. Add 0.005 gram sodium ammonium hydrogen phosphate, dissolved in a little water, agitate well, allow precipitate to settle twenty-four hours, and filter before using.

DISSOLVED OXYGEN.

Several methods of different types have been suggested for this determination; Winkler's method is preferred by the APHA. Hale and Melia have made investigations of it and give the following conclusions:

It is very easy, rapid and accurate. Duplicate samples in routine work ordinarily check within 0.1 ppm. oxygen.

Nitrite as present in the usual run of waters has no appreciable effect upon the accuracy of the results.

Nitrite in quantities upward of 0.2 ppm. increases the results by a catalytic reaction, increasing with increasing amounts of nitrite.

The effect of high nitrite present in any amount ever occurring in water may be counteracted by the use of potassium acetate solution (or sodium

acetate crystals) to neutralize the hydrochloric acid before exposure to the air. For the Hale-Melia procedure for this case, see after the general procedure.

Samples for oxygen may be taken and transported elsewhere for titration in ground glass stoppered bottles with part of the chemicals added, either in alkaline or acid condition, if kept out of contact with air. The alkaline condition is preferable, since changes in temperature can not change results, all the oxygen being in the precipitate. In either condition if air leaks in it may increase results, in alkaline condition by direct absorption, in hydrochloric acid condition by catalytic action of nitrite. It is not advisable to add the acetate until ready for titration.

Samples for oxygen may best be transported elsewhere for determination by using medicine pipets. If samples are not saturated with oxygen, the bulbs should be collapsed and samples transported without ice; if supersaturated the bulbs should be full of the water and samples iced.

Reagents:

Manganous Sulfate Solution.—Dissolve 48 grams of manganous sulfate in 100 c.c. of distilled water.

Alkaline Iodid Solution.—Dissolve 360 grams of sodium hydroxid and 100 grams of potassium iodid in 1000 c.c. of water.

Diluted Sulfuric Acid.—Strong sulfuric acid mixed with an equal volume of water. The sp. gr. will be about 1.4.

Sodium Thiosulfate Solution.—Dissolve 42 grams pure re-crystallized sodium thiosulfate in 1000 c.c. of water. This is an $^{N}/_{40}$ solution, 1 c.c. of which is equivalent to 0.002 gram of oxygen (0.1395 c.c. at 0° and 760 mm.). As the solution does not keep well, a solution of potassium dichromate of similar titer should be kept for frequent valuation. The keeping qualities of the thiosulfate solution are materially improved, by adding, for each 1000 c.c., 5 c.c. of chloroform and 1.5 grams of ammonium carbonate before making up to the prescribed volume.

Starch Solution.—A small amount of good starch is stirred with cold water until a thin paste is produced and the mixture is then poured into about 200 times its volume of boiling water, boiled for a few minutes and sterilized. It may be preserved by addition of a few drops of chloroform.

Process:

Bottles of known capacity are used, and the samples collected with precautions not to involve absorption of air. To the contents of such a bottle, with as little admission of air as possible, add 2 c.c. each of the manganous sulfate and

alkaline iodid solutions, dipping the mouth of the pipet below the surface of the liquid, close the bottle, shake, allow to settle, add 2 c.c. of diluted sulfuric acid, and mix well. These manipulations should be carried on at the time and place at which the sample is collected, but after the addition of the acid the mixture may be transported to the laboratory.

The contents of the bottle are rinsed into a flask and titrated with sodium thiosulfate solution, adding, when the liquid becomes faintly yellow, a few c.c. of starch solution as an indicator.

Hale and Melia recommend the following procedure for waters rich in nitrites: Add, at the bottom of the bottle, 2 c.c. of the manganous sulfate solution, followed by 2 c.c. of the alkaline iodid solution. Shake, allow to settle, introduce to the bottom of the liquid 2 c.c. of concentrated hydrochloric acid and shake until the precipitate is entirely dissolved. Then add 2 c.c. of potassium acetate solution (100 grams to 100 c.c.). Shake, allow to settle, add at the bottom 2 c.c. concentrated hydrochloric acid and shake until the precipitate is entirely dissolved. Add 2 c.c. potassium acetate solution (100 grams) at the bottom and mix. Withdraw by pipet 100 c.c. into an Erlenmeyer flask and titrate with $^{N}/_{10}$ thiosulfate, adding a little starch solution at the

end. Correction must be made for 6 c.c. of water displaced by the first two solutions and by the acetate solution. The acid needs no correction, since it displaces only water from which oxygen has been removed.

Precautions must be taken to exclude contact with air as much as possible until the solution is ready to titrate by replacing the stopper as quickly as possible after each reagent is introduced; to get complete solution of the precipitate by the hydrochloric acid; to give the thiosulfate a little more time to react at the end point in the acetic acid condition; to use small amounts of starch solution. For introducing the solutions it is advisable to use pipets with two marks, measuring 2 c.c., well up on the pipet so as to give head and not contaminate the upper liquid in the bottle more than necessary while actually displacing liquid from the bottle in proportion to the amount introduced.

The amount of oxygen is best stated in parts per million by weight, but it is sometimes desired to give it in c.c. for a given volume of the water. The following formulas, given by the APHA, are used in which n = number of c.c. of thiosulfate solution used, and v = the capacity of the bottle, less 4, deducted for the displacement by the reagent.

$$\frac{200n}{v} = \text{ppm. of oxygen in sample.}$$

$$\frac{139.5n}{v} = \text{c.c. of oxygen at } 0^\circ \text{ and } 760 \text{ mm., in } 1000 \text{ c.c. of sample.}$$

HARDNESS.

Water containing notable amounts of calcium and magnesium carbonates is termed "hard," the practical importance of the condition being mainly the liability to form scale in boilers, and to interfere with the formation of lather with soap; in fact, the soap-consuming power is usually intended by the term. Hardness may be caused by many substances, but the common causes are calcium and magnesium carbonates and sulfates, occasionally the chlorids and, rarely, the other halids. Magnesium sulfate is freely soluble in water, calcium sulfate slightly so, but the carbonates are almost insoluble. Natural waters, however, generally contain notable amounts of carbonic acid, and this has a marked solvent action on the carbonates. When waters thus charged with carbonates are boiled or freely exposed to the air, much of the carbonic acid is driven of, the carbonates precipitate and are not re-dissolved on cooling. On the other hand, the sulfates and chlorids are not precipitated by boiling or ex-

posure to air, hence chemists distinguish between "temporary" and "permanent" hardness, the former being that due to carbonates held in solution by carbonic acid. The APHA proposes the term "non-carbonate" as a substitute for "permanent" in this connection. Hardness may be determined in several ways. A gravimetric analysis will give accurately the substances present, but it is too tedious for ordinary routine. The amount of soap required to make a persisting lather with a given volume of the water may be measured; titration with standard acid will determine the carbonates, and, by a simple modification, the sulfates (and halogen salts) may be ascertained. Both these methods are much in vogue.

TITRATION METHOD.—This is due to Hehner, whose procedure with slight modifications is still in use. The solutions are:

$^{N}/_{50}$ *Sodium Carbonate.*—1.062 grams pure dry sodium carbonate dissolved in 1000 c.c. of water.

$^{N}/_{50}$ *Sulfuric Acid.*—$^{N}/_{10}$ acid is diluted with four times its volume of water.

Temporary Hardness (APHA Procedure)—100 c.c. of the sample are placed in a porcelain basin, 0.5 c.c. of lakmoid solution (0.1 gram to 50 c.c. of 50 per cent. alcohol) added and the sulfuric acid run in until the amount is close to the neutralizing point. The liquid is then heated

until bubbles of steam just begin to break on the surface, the heat is withdrawn and titration continued until a drop of the acid produces no change in sinking in the liquid. The number of c.c. used multiplied by 10 gives the parts per million of alkalinity in terms of calcium carbonate.

Erythrosin G (iodeosin G, sodium salt of tetriodofluorescein) is preferred by many analysts as an indicator. R. Haines has furnished some data on the subject. The common erythrosins, such as B and BB, used for staining, are unsuited on account of their fluorescence which confuses the end point. Methyl orange is disapproved by the APHA. If used, it should be in very dilute solution and not above 40°. Erythrosin is applied by the APHA as follows:

100 c.c. of the sample are placed in a bottle or flask holding 250 c.c., 2 c.c. of erythrosin solution (1 gram to 1000 c.c. of pure water) and 5 c.c. of chloroform added, and the liquid titrated with the sulfuric acid, shaking well between each addition. The rose tint of the indicator gradually diminishes and is finally discharged by a drop or two of the acid, which is the end point. The chloroform must be neutral to the indicator. The data have the same significance as with lakmoid.

Permanent (Non-carbonate) Hardness.—Hehner's method is still much in vogue, with some modifi-

cations: To 100 c.c. of the sample, sufficient of the sodium carbonate solution is added to convert the calcium and magnesium present into carbonates. Generally, 1 c.c. for each 10 parts per million of solids in the water will be satisfactory. The mixture is evaporated to dryness, the residue extracted with a little recently boiled distilled water, filtered—using a very small filter—and washed with small portions of water several times. The filtrates are mixed and titrated hot with the acid. The c.c. of acid used are deducted from the c.c. of carbonate solution added; the remainder is the parts per million of non-carbonate hardness.

Owing to the liability of glass and porcelain to yield alkali, the evaporation is best conducted in platinum or nickel, but Jena glass may in many cases be used.

If the water contains sodium or potassium carbonate, there is no permanent hardness, and more acid will be required for the filtrate than the equivalent of the sodium carbonate added. From this excess the quantity of sodium carbonate in the water may be determined.

In such case, since any alkali carbonate in the water would be erroneously calculated as temporary hardness by the direct titration, the equivalent, in terms of calcium carbonate, of the alkali carbonate present should be deducted from

the figure given by the first titration in order to get the true temporary hardness.

APHA *Method for Non-carbonate Hardness.*—This, devised by Pfeifer and Wartha, employs for precipitating the calcium and magnesium a so-called "soda-reagent," equal volumes of $^{N}/_{10}$ sodium hydroxid and sodium carbonate.

200 c.c. of the sample are placed in a Jena flask, boiled ten minutes, to expel carbonic acid, and 25 c.c. of the soda-reagent added. The mixture is concentrated to 100 c.c., cooled, transferred to a 200-c.c. flask, made up to the mark with boiled distilled water, and filtered (dry filter). The first 50 c.c. are rejected; 100 c.c. of the next portions are titrated with standard acid using erythrosin G as an indicator.

The APHA recommends $^{N}/_{20}$ acid, and with this the formula for calculation of results is $x = 12.5 (s - 2n)$ in which s is the c.c. of acid equivalent to the soda-reagent used, n the c.c. required for the titration, and x the non-carbonate hardness in parts per million of calcium carbonate. This formula can easily be adapted for any other strength of acid.

Total hardness may also be determined by this method. 200 c.c. of the sample are mixed with just sufficient standard acid to neutralize the alkalinity. The carbonates are converted into sulfates. The mixture is boiled to 100 c.c.,

cooled, mixed with 25 c.c. of soda-reagent, again concentrated to 100 c.c., cooled, made up to 200 c.c., titrated with acid and computed as above. The non-carbonate hardness is obtained by subtracting the alkalinity from the total hardness.

SOAP METHOD.—Many chemists estimate hardness by the use of soap solution, but the procedure, at best, is inferior in accuracy. The following is essentially the method given by the APHA.

Calcium Chlorid Solution.—0.2 gram pure dry calcium carbonate is dissolved in a little pure dilute hydrochloric acid, care being taken to avoid loss by spattering. The liquid is evaporated to dryness, re-dissolved in a little water and again evaporated, this operation being repeated several times to ensure the removal of the excess of acid. The residue is dissolved finally and made up to 1000 c.c. 1 c.c. = 0.0002 gram calcium carbonate.

Soap Solution.—Dissolve 25 grams of fine white Castile soap in 250 c.c. of 80 per cent. alcohol. Allow the solution to stand several days, decant a convenient quantity and dilute with 70 per cent. alcohol to such a volume that 6.4 c.c. will just make a permanent lather with 20 c.c. of the calcium chlorid solution, following the technic described below. Usually from 75 to 100 c.c. of the strong solution will be required to make 1000 c.c. of the dilute standard. The

latter solution must be titrated from time to time with the calcium chlorid solution to detect any change of ratio.

The term "Castile" as applied to soap has now no definite value; any manufacturer may use it. It is best, therefore, to use commercial oleic acid now obtainable of good quality and saponify cautiously with good sodium hydroxid.

The procedure with soap solution is as follows: 50 c.c. of the sample are placed in a 250-c.c. flask, and the soap solution added in portions of not more than 0.3 c.c. at a time, shaking well between each addition. When a lather forms over the whole surface of the water and remains substantially unchanged for five minutes while the flask lies on its side, the buret should be read.

The soap solution must be added in small portions, especially in presence of notable amounts of magnesium compounds. If much carbonic acid is present it is an advantage to remove some of it by suction with a filter pump, taking care, of course, not to cause precipitation of carbonates. If the sample requires more than 7.9 c.c. of soap solution, a smaller volume, 25 c.c., 10 c.c., sometimes even 2 c.c., must be diluted with recently boiled distilled water and the test repeated, but the results in such cases will be less accurate.

Degrees of Hardness.—Hardness tests are often reported under this term, which should be

abandoned. The standards differ in different nations.

English degrees (*Clark's scale*) are the equivalent in grains per Imperial gallon, and must be multiplied by 14.3 to convert them to parts per million.

French degrees represent parts per 100,000 and are converted to parts per million by multiplying by 10.

C.c. Soap Solution	Hardness	C.c. Soap Solution	Hardness	C.c. Soap Solution	Hardness
0.8	1.6	3.2	35.1	5.6	68.6
0.9	3.2	3.3	36.4	5.7	70.0
1.0	4.8	3.4	37.7	5.8	71.4
1.1	6.3	3.5	38.0	5.9	72.9
1.2	7.9	3.6	40.3	6.0	74.3
1.3	9.5	3.7	41.6	6.1	75.7
1.4	11.1	3.8	42.9	6.2	77.1
1.5	12.7	3.9	44.3	6.3	78.6
1.6	14.3	4.0	45.7	6.4	80.0
1.7	15.6	4.1	47.1	6.5	81.4
1.8	16.9	4.2	48.6	6.6	83.9
1.9	18.2	4.3	50.0	6.7	84.3
2.0	19.5	4.4	51.4	6.8	85.7
2.1	20.8	4.5	52.9	6.9	87.1
2.2	22.1	4.6	54.3	7.0	88.6
2.3	23.4	4.7	55.7	7.1	90.0
2.4	24.7	4.8	57.1	7.2	91.4
2.5	26.0	4.9	58.6	7.3	92.9
2.6	27.3	5.0	60.0	7.4	94.3
2.7	28.6	5.1	61.4	7.5	95.7
2.8	29.9	5.2	62.9	7.6	97.1
2.9	31.2	5.3	64.3	7.7	98.6
3.0	32.5	5.4	65.7	7.8	100.0
3.1	33.8	5.5	67.1	7.9	101.5

German degrees represent parts per 100,000 of

calcium oxid, and are to be multiplied by 17.8 to obtain parts per million of calcium carbonate.

Of course, degrees of either type may be obtained by dividing the figure for parts per million by the above factors respectively.

The table on page 54 gives the parts per million of calcium carbonate, or its equivalent in soap-precipitating power, corresponding to the amount of soap solution used.

POISONOUS METALS.

Under this conventional title are included *barium*, *chromium*, *zinc*, *arsenic*, *copper*, and *lead; manganese*, *iron*, *aluminum*, also, though not usually classed in this group, are objectionable when present in notable amounts.

In the commercial laboratory, in which water samples from many different places are examined, it is convenient to have a routine general test for metals precipitable as sulfids. This is most conveniently done by adding to a small volume of the water, in a colorless glass vessel, a drop or two of ammonium (or sodium) sulfid. A distinct precipitate, unless due to iron, will indicate a poisonous metal. A mere opalescence may be neglected, as it is due to separated sulfur. The iron present in ordinary waters is not usually sufficient to give a precipitate. The water must

not be acid when making the test. Care must be taken not to mistake white zinc sulfid for the sulfur precipitated by the oxidizing action of the air in the water on the alkaline sulfid.

Barium is rarely present, and only in water containing no sulfates. It can be detected by slightly acidifying the water with hydrochloric acid, filtering if necessary, and adding solution of calcium sulfate. The precipitated barium sulfate may be collected and weighed.

Chromium is rarely present, but may be looked for in the waste waters of dye-works and similar sources. To detect it, a considerable volume of the water is evaporated to dryness with addition of a small amount of potassium chlorate and nitrate, transferred to a porcelain crucible and brought to quiet fusion; any chromium present will be found in the residue in the form of chromate. The fused mass, after cooling, is boiled with a little water, filtered, the filtrate rendered slightly acid with hydrochloric acid, and a solution of hydrogen dioxid added. In the presence of chromium a transient blue color will appear; by adding a little ether and shaking the mixture, the color will pass into the ether, and on standing will form a blue layer on the surface of the water.

Zinc is best detected by the test described by Allen. The water is rendered slightly alkaline by addition of ammonium hydroxid, heated to boil-

ing, filtered, and the clear liquid treated with a few drops of potassium ferrocyanid; in the presence even of the merest trace of zinc a white precipitate will be produced.

Arsenic is most readily detected by Reinsch's test. 1000 c.c. of the water is rendered slightly alkaline by pure sodium carbonate, and evaporated nearly to dryness in a porcelain basin. 2 or 3 c.c. of water strongly acidulated with hydrochloric acid are placed in a small test-tube, about ½ of a square centimeter of bright copper foil is added, and the liquid boiled gently for a few moments. If the copper remains bright, showing that the reagents contain no arsenic, the water-residue is acidified with hydrochloric acid, added to the contents of the test-tube, and the liquid again boiled for several minutes. If arsenic be present, a steel-gray stain will appear on the copper. The slip is removed, washed with distilled water, *thoroughly* dried by pressure between filter paper, inserted into a narrow glass tube closed at one end, which has been previously dried by heating nearly to redness. The tube is gently heated at the point at which the copper rests; the deposit will sublime and collect on the cooler portion of the tube, in crystals which the microscope shows to be octahedral.

Since small amounts of arsenic frequently occur in reagents and in glass vessels, care must

be taken to avoid such sources of error. Sodium carbonate solution may contain arsenic dissolved from the glass bottle in which it is kept. It is best, therefore, to use the solid carbonate for rendering the water alkaline, and to determine its purity before use.

Iron is detected by the addition of a drop of ammonium sulfid to the water in a tall, glass cylinder. Ferrous sulfid is formed, having a greenish-black color, instantly discharged by acidifying the water with dilute hydrochloric acid. A still better test is the production of a blood-red color, with potassium thiocyanate, due to the formation of ferric thiocyanate. The water should be first boiled with a few drops of nitric acid, to convert the iron to the ferric condition, cooled, and a drop or two of the solution of potassium thiocyanate added. The test is very delicate.

DETERMINATION.—The following method was devised by Thompson and is described in Sutton's "Volumetric Analysis:"

Standard Ferric Sulfate.—0.7 gram ferrous ammonium sulfate is dissolved in water acidified with sulfuric acid, and potassium permanganate solution added until the solution turns a very faint pink color. The solution is diluted to 1000 c.c. 1 c.c. contains 0.1 milligram iron.

Diluted Nitric Acid.—30 c.c. concentrated nitric acid diluted with water to about 100 c.c.

Potassium Thiocyanate.—5 grams of the salt dissolved in about 100 c.c. water.

100 c.c. of the water are evaporated to small bulk, acidified with hydrochloric acid, and just sufficient dilute potassium permanganate solution added to convert all the iron to the ferric condition. The liquid is evaporated nearly to dryness to drive off excess of acid, then diluted to its original volume, 100 c.c. In two tall glasses marked at 100 c.c., 5 c.c. of the nitric acid and 15 c.c. of the thiocyanate solution are placed. To one of these a measured volume of the treated water is added and both vessels filled up to the mark with distilled water. If iron is present, a blood-red color will be produced. Standard iron solution is added to the second vessel until the color agrees. The amount of water which is added to the first glass will depend upon the quantity of iron it contains; not more should be used than will require 2 or 3 c.c. of the standard to match it, otherwise the color will be too deep for comparison.

Manganese.—The following method is described by Wanklyn in his treatise on water analysis. About 1000 c.c. of the water is evaporated to small bulk, nearly neutralized by hydrochloric acid and treated with a few drops of a solution

of hydrogen dioxid. The formation of a brown precipitate indicates the presence of manganese. The test is very delicate. The precipitate may be collected on a filter, the filter ashed, and the residue fused with a mixture of sodium carbonate and potassium nitrate. Green potassium manganate will be produced, which, when boiled with water, will give a bright-red solution of potassium permanganate. The quantitative determination is given elsewhere.

Aluminum.—Traces of this element are to be expected in all waters, and it is not usual to test for it except in elaborate analysis of the mineral ingredients, as described in another section. The use of aluminum sulfate as a coagulant in many rapid-filtration methods makes it necessary to examine effluents for excess of precipitant. As the precipitating power depends on the reaction with carbonates, the alkalinity of the water is diminished in proportion to the amount of aluminum compounds decomposed, hence if the latter is in excess the water will be acid to many indicators. Quantitative tests may be made by titrating with lakmoid, phenacetolin, or erythrosin G. Methyl orange is not suitable. Leffmann has found that sodium alizarin-monosulfonate has a special action in the presence of excess of aluminum compounds. This indicator is red when alkaline, yellow when acid, but in water containing

aluminum sulfate it assumes an intermediate tint which a moderate excess of mineral acid does not affect.

Lead may be readily detected by adding to the water in a tall glass cylinder a drop of ammonium sulfid; brownish-black lead sulfid is formed, which does not dissolve either by acidulating the water with dilute hydrochloric acid (distinction from iron), nor by the addition of about 1 c.c. of a strong solution of potassium cyanid (distinction from copper). As a delicate test, S. Harvey gives the following method: 250 c.c. of the sample are placed in a precipitating jar, about 0.1 gram of crystallized potassium dichromate is added and dissolved by agitation. The same volume of lead-free water is treated in the same manner, and the two liquids compared side by side. Water containing 0.3 part per million will show a turbidity in fifteen minutes which will be rendered more distinct by contrast with the clear water alongside. By allowing the jar to stand for about twelve hours undisturbed, the precipitate will settle and will become still more distinct. No other metal likely to be present in water will give a similar reaction.

In the absence of copper, lead may be determined as follows: A solution is prepared containing 0.16 gram of lead nitrate to 100 c.c.; 1 c.c. of this contains 0.001 gram lead. 100 c.c. of the

water to be tested are placed in a tall glass vessel, made acid by the addition of a few drops of acetic acid, and 5 c.c. of hydrogen sulfid added. In a similar vessel 100 c.c. of distilled water are placed, together with the same quantities of acetic acid and hydrogen sulfid, and sufficient of the standard lead solution to match the tint in the first cylinder. The amount of lead in the water under examination is thus known.

Copper is detected in the same manner as lead by acidifying the water with acetic acid and adding hydrogen sulfid. The precipitate is distinguished from lead sulfid by the fact that the color is discharged on the addition of about 1 c.c. of a strong solution of pure potassium cyanid. It may be further confirmed by the addition to another portion of the water of a solution of potassium ferrocyanid. In the presence of even a very small amount of copper, a mahogany-red is produced.

In the absence of lead, copper is determined in the same way as that metal, using a standard solution of copper for the comparison liquid, made by dissolving 3.929 grams of crystallized copper sulfate in 1000 c.c. of water. 1 c.c. of the solution contains 1 gram copper.

H. C. Bradley states that hematoxylin (from logwood) is a delicate test for copper. It is best used in weak alcoholic solution to which a trace

of sodium hydroxid has been added, which renders the liquid faintly pink. A few drops of this reagent will in the course of a few minutes give a distinct blue even with very small amounts of copper. Moderate amounts produce a precipitate. The liquid should stand for an hour or so before deciding that the result is negative. Free mineral acid interfere with the test.

If both lead and copper are present, a large quantity of the water should be evaporated to small bulk, and the metals separated by one of the standard laboratory methods.

As copper sulfate is much used to prevent growth of algæ, it is advisable to test for it in surface waters.

GENERAL QUANTITATIVE ANALYSIS.

Silica, Iron, Aluminum, Manganese, Calcium, and Magnesium.—A considerable volume of the sample (in case of water containing only moderate amounts of solids, at least 1000 c.c.) is evaporated to small bulk, rendered acid with dilute hydrochloric acid, the evaporation carried to dryness, the residue heated briefly to 180°, or baked for thirty minutes at about 110° on an asbestos plate, treated with a little dilute hydrochloric, again evaporated to dryness, some strong hydrochloric acid added, diluted, the mixture filtered, and the

separated *silica* washed, dried, ignited in a platinum crucible, and weighed.

To the filtrate, previously boiled with a few drops of strong nitric acid, excess of ammonium hydroxid is added, the liquid boiled several minutes, the precipitate collected, washed with boiling water, dried, ignited, and weighed. It consists of Fe_2O_3 and Al_2O_3; also all the phosphates and some manganese if much of the latter is present in the water. In such cases the precipitate before drying is redissolved in hydrochloric acid and neutralized with a dilute solution of ammonium carbonate until the water becomes almost turbid. It is then boiled, and the precipitate, now free from manganese, washed, dried, ignited, and weighed. The iron may be determined by dissolving the precipitate in strong hydrochloric acid and employing the colorimetric method described on page 58.

If no manganese, or only traces are present, the filtrate from the ammoniacal solution is heated to a moderate temperature, and strong solution of ammonium oxalate added in small portions with stirring, until the precipitate tends to settle readily and leave the liquid clear. The precipitate is collected on a filter and washed. It is calcium oxalate only, if but little magnesium is present in the water, but if the proportion of magnesium is large some magnesium oxalate may be

also thrown down. In this case, the precipitate should be dissolved in a minimum quantity of hot strong hydrochloric acid, the solution diluted with water, excess of ammonium hydroxid and a small quantity of ammonium oxalate. After the precipitate has completely subsided, it is collected on a filter, washed and dried. If quite small in amount, it is placed with the filter in a weighed platinum crucible, ignited over the Bunsen burner for a short time, and then over the blast lamp for from five to fifteen minutes. Calcium oxid is thus obtained; it is allowed to cool in the desiccator and weighed. The weight multiplied by 0.715 gives the weight of *calcium*. When the amount of precipitate is large, it is better to remove it from the filter, and heat it just short of redness until it assumes a grayish tint. It then consists of calcium carbonate. To this is added the ash of the filter. The weight of the calcium carbonate multiplied by 0.4 gives the weight of *calcium*.

Calcium may be also determined by titration. The precipitate of calcium oxalate, after thorough washing, is dissolved from the filter by warm, dilute, sulfuric acid, heated to about 65°, and titrated with decinormal permanganate until a pink tint is obtained. 1 c.c. of the permanganate is equivalent to 0.002 gram calcium; 0.0028 calcium oxid; 0.0050 calcium carbonate.

The filtrates from the calcium oxalate are mixed, slightly acidified with hydrochloric acid, concentrated and cooled, ammonium hydroxid and sodium phosphate added in excess, stirred briskly and allowed to stand in the cold for about twelve hours. The precipitated ammonium magnesium phosphate is brought upon a filter, that adhering to the sides of the vessel being dislodged by rubbing with a glass rod tipped with a piece of clean rubber tubing. It is washed with a solution made by mixing 1 part of the ammonium hydroxid of 0.96 sp. gr. with 3 parts of water. The precipitate is dried, transferred to a platinum crucible, the filter ashed separately and added to it, and the whole heated at first gently and then to intense redness for several minutes. After cooling, it is weighed. It consists of magnesium pyrophosphate; the weight multiplied by 0.218 gives the weight of *magnesium*.

Manganese, if present in appreciable quantity, is separated before the precipitation of the calcium, as follows: The filtrate from the iron precipitate is slightly acidulated with hydrochloric acid, concentrated, and the manganese precipitated as sulfid by colorless or slightly yellow solution of ammonium sulfid. The flask, which should be nearly full, is stoppered, allowed to rest in a moderately warm place until the precipitate has thoroughly settled, filtered, washed with

dilute ammonium sulfid water, and purified by dissolving in a small quantity of hydrochloric acid and reprecipitating with ammonium sulfid. It is filtered off, washed as before, dried, placed in a weighed porcelain crucible, covered with a little sulfur, and ignited in a current of hydrogen introduced into the crucible by a tube passing through a hole in the cover. The pure manganese sulfid thus obtained is allowed to cool and weighed. The weight multiplied by 0.633 gives *manganese*.

Sulfates.—500 c.c. of the clear water are slightly acidulated with hydrochloric acid, heated to boiling, and barium chlorid solution added in moderate excess. The precipitated barium sulfate is allowed to subside completely, collected upon a filter, washed thoroughly, dried, and incinerated. The weight multiplied by 0.411 gives SO_4. If the proportion is very low, it will be advisable to concentrate the water to much smaller bulk before precipitating.

Control. Potassium, Sodium, and Lithium.—From 250 to 1000 c.c. of the water, according to the amount of solid matters present, are evaporated to dryness in a platinum dish, and the residue treated with a small amount of water and sufficient dilute sulfuric acid to decompose the salts present. The dish should then be covered and placed upon the water-bath for ten minutes, after which any liquid spurted on the cover is

washed into the dish, the whole evaporated to dryness and heated to redness. A few drops of ammonium carbonate solution should then be mixed with the residue, and the ignition repeated to insure the removal of the last portions of free acid. In the majority of cases the only basylous elements present in considerable quantity are calcium, magnesium, and sodium. The *sodium* may be determined indirectly, therefore, by calculating from the amount of calcium and magnesium found, the calcium and magnesium sulfate in the residue, and subtracting this sum, together with the silica, from the total residue.

For the determination of potassium and sodium in ordinary well and river waters, not less than 2 liters should be employed. When lithium is to be determined, it is generally necessary to use much more. In any case, as the alkalies are to be weighed as chlorids, it is advisable, if notable amounts of sulfates are present, to precipitate them by addition of barium chlorid.

The water is evaporated to about 200 c.c., a slight excess of calcium hydroxid added to the hot liquid—generally 3 c.c. of thin milk of lime will be sufficient—and the heat continued for several minutes. It is then washed into a 250-c.c. flask, disregarding the insoluble portion adhering to the dish, which, however, should be thoroughly washed, and the washings added to the flask.

After cooling, the flask is filled up to the mark with distilled water, thoroughly mixed, the precipitate allowed to settle, and the liquid filtered through a dry filter. 200 c.c. of the filtrate are measured into another 250 c.c. flask, ammonium carbonate and ammonium oxalate added, filled with water up to the mark, mixed, allowed to settle, filtered through a dry filter, 200 c.c. of the filtrate measured off, and evaporated to *thorough dryness* in a platinum crucible, heating very cautiously at the last stages, to avoid loss by spurting. The low-temperature burner is suited for this purpose. The crucible is now covered and cautiously heated to dull redness, cooled, and weighed. The residue contains the potassium, lithium, and sodium as chlorids. It contains, sometimes, also, traces of magnesium, which may be removed by treating again with lime and ammonium carbonate and oxalate. It is frequently of advantage, in evaporating these saline solutions, to add, when the solution becomes concentrated, several c.c. of strong hydrochloric acid. This precipitates the greater portion of the salts in a finely granular condition, and renders loss by spurting less liable to occur.

If potassium and sodium chlorids only are present, the residue is dissolved in a small quantity of water, an excess of a concentrated

neutral solution of platinum chlorid added, evaporated to small bulk at a low heat on the water-bath, some 80 per cent. alcohol added, allowed to stand, the clear liquid decanted off on a small filter, and the residue washed in this way several times by fresh, small portions of the alcohol. The precipitate is then washed on to the filter with alcohol, washed again with the alcohol, thoroughly dried and transferred as far as possible to a watch-glass. The small portion on the filter is dissolved off and the solution placed in a weighed platinum dish and evaporated to dryness. The main portion on the watch-glass is then added, and the whole dried to a constant weight at about 125°, cooled, and weighed. The weight thus found multiplied by 0.3 gives the weight of *potassium chlorid.* This subtracted from the combined weight of the chlorids gives the weight of *sodium chlorid.*

Lithium, if present, is best separated before the treatment with platinum chlorid. The following method, devised by Gooch, gives good results: To the concentrated solution of the weighed chlorids, amyl alcohol is added and heat applied, gently at first, to avoid bumping, until the water disappears from the solution and the point of ebullition becomes constant at a temperature which is approximately that at which the alcohol boils (132°), the potassium

and sodium chlorids are deposited, and the lithium chlorid is dehydrated and taken into solution. The liquid is then cooled, and a drop or two of strong hydrochloric acid added to reconvert traces of lithium hydroxid in the deposit and the boiling continued until the alcohol is again free from water. If the amount of lithium chlorid be small, it will be found in the solution and the potassium chlorid and sodium chlorid in the residue, excepting traces which can be allowed for. If the lithium chlorid exceed 10 or 20 milligrams, the liquid may be decanted, the residue washed with amyl alcohol, dissolved in a few drops of water, and treated as before. For washing, amyl alcohol, previously dehydrated by boiling, is to be used and the filtrates are to be measured apart from the washings. In filtering, the Gooch filter with asbestos felt may be used with advantage, applying gentle pressure by the aid of the filter-pump. The crucible and residue are ready for weighing after gentle heating over the low-temperature burner. The weight of the insoluble chlorids is to be corrected by adding 0.00041 for every 10 c.c. of amyl alcohol in the filtrate, exclusive of the washings, if only sodium chlorid be present; 0.00051 for every 10 c.c. in only potassium chlorid, and 0.00092 in the presence of both these chlorids.

The filtrate and washings are evaporated to dryness in a platinum crucible heated with sulfuric acid, the excess driven off, and the residue ignited to fusion, cooled, and weighed. From the weight is to be subtracted, for each 10 c.c. of filtrate, 0.0005, 0.00059, or 0.00109, according as only sodium chlorid, potassium chlorid, or both were present in the original mixture.

Hydrogen Sulphid.—The following is the process recommended by the APHA.

Standard Iodin Solution.—1.269 grams of pure dry iodin and 1.8 grams of pure potassium iodid are dissolved in about 50 c.c. of water and the solution made to 1000 c.c. It is $^{N}/_{100}$.

Standard Thiosulfate Solution.—2.5 grams of pure sodium thiosulfate are dissolved in water and made up to 1000 c.c. This is approximately $^{N}/_{100}$ and its relation to the iodin solution should be ascertained frequently, as it does not keep well even in the dark.

Starch Solution.—See p. 44.

10 c.c. of the iodin solution are placed in a dry 1000 c.c. flask, 1000 c.c. of the sample added, the flask well closed, shaken and allowed to stand for several hours. The excess of iodin is determined as usual by titrating with thiosulfate and starch. 1 c.c. of iodin solution is equivalent to 0.00017 hydrogen sulfid.

Free and Half-bound Carbonic Acid.—Several methods for the determination of these data have been devised. Pettenkofer's is much used but Forbes and Pratt, after careful study of different methods, favor the Lunge-Trillich (also termed the Seyler) method, which they describe as follows:

100 c.c. of the sample are placed in a tall glass cylinder, by means of a siphon (in order to avoid contact with air), 6 drops of neutral alcoholic solution of phenolphthalein added, and $^{N}/_{50}$ sodium carbonate run in from a buret with careful stirring, until a faint permanent pink is obtained. If the water contains much free carbonic acid, it is better to take less than 100 c.c., and in every case care must be taken not to stir the sample so vigorously as to cause loss of the acid, nor to proceed so slowly that it may be absorbed from the air. The solutions must be carefully standardized and preserved so that they do not absorb the acid. The fixed carbonic acid is determined on another portion of the sample by Hehner's method as given on p. 48. In waters acid to phenolphthalein this will be equal to the half-bound.

When the water is alkaline to phenolphthalein, the alkalinity with this indicator is first determined, then the total alkalinity by Hehner's method. Twice the phenolphthalein alkalinity subtracted from the total alkalinity gives the

half-bound acid, no free acid being present in this case, and the half-bound being less than the fixed. If the water is neutral to phenolphthalein, the half-bound may be equal to the fixed.

Forbcs and Pratt have modified the Pettenkofer methods as follows:

A glass-stoppered bottle, holding approximately 480 c.c., is accurately calibrated by weighing completely filled with water. It is filled with the water to be analyzed by means of a siphon, the glass stopper inserted, leaving no air-bubble, and the neck of the bottle wiped dry. The glass stopper is then carefully removed, and the 57 c.c. of the water withdrawn by means of an accurately calibrated pipet, in order to make room for the reagents. 3 c.c. of barium chlorid solution (8 grams per 1000 c.c.), 2 c.c. of saturated ammonium chlorid solution, and 50 c.c. of standard barium hydroxid are then introduced, the bottle quickly stoppered, well shaken, and set aside to settle.

There is now in the bottle an air space of only 2 c.c., which is left to avoid the possibility of loss of liquid when the stopper is inserted. After the precipitated carbonates have completely settled, several portions of 100 c.c. are siphoned off and titrated with $^{N}/_{50}$ sulfuric acid, against which the barium hydroxid is standardized. Leffmann and Beam used barium hydroxid

approximately $^{N}/_{15}$, preserved out of contact with the air, the bottle being fitted with an arrangement whereby the air was drawn through soda-lime before entering either the bottle or the buret. The figure obtained by averaging several results of titration of portions of 100 c.c. is taken as the true value.

The use of this large quantity of water and the titration of 100 c.c. portions reduce considerably the errors due to the difficulty of obtaining the exact end-point, and those due to inaccuracies of measurement.

Boric Acid.—To detect this add to a considerable volume of the water sufficient sodium carbonate to render it distinctly alkaline; evaporate to dryness, acidify with hydrochloric acid, moisten a slip of turmeric paper with the liquid, and dry it at a moderate heat. In the presence of boric acid the paper will assume a distinct *brown-red.*

ANALYSIS OF BOILER SCALE.

If the scale is made up of pieces of decidedly different quality, some being hard and gritty, others soft and friable, separate tests should be made on representative samples of each sort; but if the general character is fairly uniform, it will be sufficient to sample the entire mass and reduce about 5 grams to a fine powder, finishing

in an agate mortar. All of the quantity selected as the sample should be equally finely powdered.

0.5 gram should be heated in a covered beaker with moderately strong hydrochloric acid, until all soluble matter is dissolved; the liquid is then evaporated to dryness on the water-bath, redissolved in water containing some hydrochloric acid, and filtered. The precipitate is silica. The filtrate and washings are mixed and divided into convenient parts. One part is used for the determination of sulfates, and the other for iron oxid, alumina, calcium, and magnesium, according to the methods given on pages 64 to 66. Scale often contains an appreciable amount of oil. This may be determined by extracting a known weight of the finely powdered material with a petroleum spirit that leaves no residue on evaporation on the water-bath.

SPECTROSCOPIC EXAMINATION.

For the ordinary spectroscopic examination of a water a simple apparatus will suffice. The arrangement shown in the cut (Fig. 5) is a small direct-vision spectroscope, held in a universal stand, with an adjustable burner as the source of heat.

For the examination a considerable volume should be evaporated nearly to dryness, a little hydrochloric acid being added near the end of the

process, the residue placed in a narrow strip of platinum foil, having the sides bent so as to retain the liquid, and heated in the flame. While this method will be sufficient in many cases, a far better plan is to separate the substance sought for in a state of approximate purity and then examine with the spectroscope. Very small traces of lithium, for instance, may be detected as follows: To about 1000 c.c. of the water sufficient sodium carbonate is added to precipitate all the calcium and magnesium, and the liquid boiled down to about one-tenth its bulk; it is then filtered, the filtrate rendered slightly acid with hydrochloric acid, and evaporated to dryness. The residue is boiled with a little alcohol, which will dissolve out the lithium chlorid. The alcoholic solution is evaporated to dryness, the residue taken up with a little water and tested in the flame.

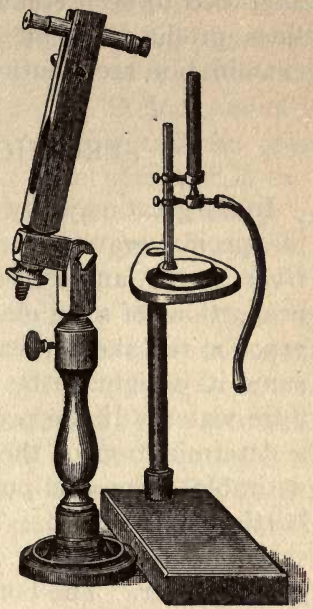

FIG. 5.

In order to identify with certainty any line which may be obtained, it is only necessary to hold in the flame at the same time a wire which has been dipped in a solution of the substance supposed to be present and to note whether the lines produced by it and the material under examination are identical.

SPECIFIC GRAVITY.

In the great majority of cases the determination of specific gravity is not essential. Ordinary river, spring, and well waters contain such small proportions of solid matter that it is usually the practice to take a measured volume and to assume its weight to be that of an equal bulk of pure water. If the proportion of solids is high, a determination of the specific gravity may be desirable. For this purpose the specific-gravity bottle may be used. This consists merely of a small flask provided with a finely perforated glass stopper. The bottle is weighed first alone, then filled with distilled water at 15.5°, and finally with the water under examination at the same temperature. In filling the bottle, the liquid is first brought to the proper temperature, the bottle completely filled, the stopper inserted, and the excess of water forced out through the perforation and around the sides of the stopper carefully

removed by bibulous paper. The weight of the water examined divided by the weight of the equal bulk of distilled water at the same temperature gives the specific gravity.

Another method, and one which gives very satisfactory results, is by the use of a plummet. This may conveniently consist of a piece of a thick glass rod of about 10 c.c. in bulk, or of a test-tube weighted with mercury and the open end sealed in the flame. The plummet is suspended to the hook of the balance by means of a fine platinum wire and its weight ascertained. It is then immersed in distilled water at 15.5°, and the loss in weight noted. The figure so obtained is the weight of a bulk of water equal to that of the plummet. This having been determined, the specific gravity of any water may be found by immersing in it the plummet and noting the loss in weight. This, divided by the loss suffered in pure water, gives the specific gravity.

ACTION OF WATER ON METALS.

The most important are actions on lead and iron. The phenomena have been extensively studied, but much remains to be determined. It may be said, as a general principle, that oxygen and carbonic acid in solution have a great influ-

ence in determining corrosion, but there seems to be no doubt that with most waters minute amounts of common metals pass into a colloidal state, and are then oxidized or transformed into various salts. Whether the metal will remain in the dissolved or colloidal condition depends, therefore, on the salts present.

Action of water on lead has great sanitary significance, as even in minute amounts lead will soon produce serious toxic effects. The action on iron is chiefly important from an industrial point of view.

It will not be necessary to give at length the results of the many investigators—results by no means always concordant—but the following are summaries of some of the observations: Crookes, Odling and Tidy found that when soft waters did not act on lead silica was present in notable amount, while with waters having marked action silica was in small amount. These results were confirmed by laboratory experiments. They also found that an effective way of silicating a water is to pass it over a mixture of flint and limestone. The reason for this was pointed out later by Carnelly and Frew, who showed that while calcium carbonate and silica both exert a protective influence, calcium silicate is more effective than either; and, further, that in almost all cases in which corrosion takes place, it is greater in the

presence of oxygen. This is particularly the case with potassium and ammonium nitrates and with calcium hydroxid. The reverse is true of calcium sulfate, which is more corrosive when air is excluded. Their experiments also show that the presence of calcium carbonate or calcium silicate altogether prevents corrosion by potassium and ammonium nitrates.

As the result of an elaborate series of experiments, Müller concludes that, while chlorids, nitrates, and sulfates all act upon lead pipes, no corrosion takes place in the presence of sodium acid carbonate, and that calcium carbonate, by taking up carbonic acid, acts in the same way. This latter conclusion is at variance with the observations of Carnelly and Frew, who found that calcium carbonate is equally effective when carbonic acid is excluded. Müller also states that surface waters, contaminated by sewage and containing large amounts of ammoniacal compounds, will dissolve lead under all circumstances.

Allen has shown that water containing free acid, including sulfuric acid, acts energetically upon lead. This is not surprising in view of the later experiments, which prove that even calcium sulfate is corrosive. Later, Carleton-Williams found that even in the presence of free acid, corrosion may be prevented by the addition of sufficient silica. His experiments also confirm

the view generally held, that soluble phosphates protect lead to a marked degree.

The above data lead to the following classification:

Corrosive: Free acid or alkalies, oxygen, nitrates, particularly potassium and ammonium nitrates, chlorids, and sulfates.

Non-corrosive and preventing corrosion by the above: Calcium carbonate, sodium acid carbonate, ammonium carbonate, calcium silicate, silica, and soluble phosphates.

All these investigations overlook the possibility of the initial action being the formation of colloidal lead, so it is likely that later experiments will throw more light on the question.

As regards the action on iron, the following data are taken from a recent report by Hale: Corrosion is rapid in new pipe: steel is most readily attacked, wrought iron next, galvanized iron the least. The chief agent in dissolving iron and holding it in solution is carbonic acid; the chief agent in rusting is oxygen. By the action of carbonic acid about 70 per cent. of the oxygen comes from the decomposition of water, hydrogen being set free; the remainder is derived from the dissolved oxygen. Soft waters containing much carbonic acid are most corrosive; hard waters cause but little complaint as a rule as the iron becomes soon coated.

The action in distribution services probably never reaches completion on account of the protecting scale of oxid. Since in distribution there is no exhaustion of oxygen, an increase of carbonic acid may mean an increase of iron in the water, and this would be precipitated in the hot water system.

Neutralization of both free and half-bound carbonic acid is inadvisable as the slightest excess of alkaline hydroxid will increase corrosion and, in hot water, increase the solution of zinc. It is also stated that this excess will interfere with the reaction with alum, with color removal and produce deposits that choke pipes and meters. Neutralization of free carbonic acid is advisable.

The corrosive action of water very poor in solids can be materially diminished by filtration through crude bone-charcoal.

In boilers, the greatest corrosion is generally observed at the point at which the cold water enters, since there the gases are driven out of solution and attack the metal.

The corrosive action of oxygen and carbonic acid in waters comparatively pure, such as those derived from mountain springs, was repeatedly observed by Dr. William Beam, in examination of waters used for the locomotives of the Baltimore and Ohio Railroad. The waters which caused

the most corrosion were mainly those containing small quantities of solid matter, the full amount of oxygen, and considerable carbonic acid, but no other acid or acid-forming body.

Free acid, other than carbonic acid, is not often found in water, and if present renders the water unfit for use, unless it be neutralized. Mine waters are the most likely to contain free acid, sulfuric acid being generally present. Sometimes the acidity is due to organic acids. These act very injuriously on iron. Allen gives an example of this in the water supplied to Sheffield, England, which he found to contain an organic acid in amount equivalent to from 3.5 to 10 parts of sulfuric acid per million.

Boiler Waters.—The main conditions affecting the value of a water for steam-making purposes are its tendency to cause corrosion and the formation of scale.

Magnesium chlorid may be harmful if in considerable quantity. At a temperature of 155°, corresponding to an effective pressure of four atmospheres, magnesium chlorid reacts with water to form magnesium oxid and hydrochloric acid, the latter attacking the boiler, especially at the water-line. If at the same time considerable calcium carbonate is present, the action may be somewhat lessened, but, as Allen has pointed out, and as Leffmann and Beam

have noticed, there may still be corrosion, so that the presence of more than a small quantity of the salt, say a grain or two to the gallon, may be considered objectionable. Allen remarks that the presence of a certain amount of sodium chlorid may prevent this decomposition, the two chlorids combining to form a stable double salt. The addition, therefore, of common salt to a water containing magnesium chlorid may act to diminish corrosion, a point which will bear further investigation.

It has not been determined how far the presence of nitrites, nitrates, and ammonia affects the quality of water for steam-making purposes; but it is more than probable that they act harmfully, especially the nitrates, which are frequently present in large amount.

Scale is composed of matters deposited from the water either by the decompositions induced by the heat or by concentration. When the deposit is loose, it is termed *sludge* or *mud*, and usually consists of calcium carbonate, magnesium oxid, and a small amount of magnesium carbonate. The magnesium oxid is formed by the decomposition of the magnesium carbonate and chlorid.

The formation of sludge is the least objectionable effect, since it may readily be removed by "blowing off," provided that care is previously

taken to allow the flues to cool down so that when the water is removed the heat of the flues may not bake the deposit to a hard mass. Waters containing calcium sulfate form hard incrustations difficult to remove and causing great loss of fuel by interfering with the transmission of the heat to the water. It not only forms a hard incrustation in itself, but becomes incorporated with the mud, and renders it also hard. The hard scale will also contain practically all the silica and the iron and aluminum present in the water, besides any matters originally held in suspension.

It follows from the above that a water only temporarily hard will, if care is taken in the management of the boiler, cause the formation merely of a loose deposit of sludge—temporary hardness being due in the main to calcium and magnesium carbonates. A water permanently hard will probably form a hard scale, since such hardness is usually due to calcium sulfate. For methods of analyzing scale, see page 75.

In accordance with these principles, the analysis of a water for steam-making purposes may include the determinations of free acid, total solid residue, SO_4, Cl, Ca, Mg, temporary and permanent (non-carbonate) hardness. In cases in which the qualitative tests show but small amounts of SO_4 and Cl, the analysis may be

limited to the determinations of the temporary and permanent hardness.

In the laboratory of the Pennsylvania Railroad an approximate determination of scale-forming ingredients is made in the following manner: The total solids obtained by evaporation are treated with 66 per cent. alcohol and the undissolved residue is denominated "scale-forming material."

It has been pointed out in an earlier chapter that it is not possible to deduce from the analytic results the exact forms in which the various elements are combined, but since it is known that at the high temperature ordinarily reached in boilers definite chemical changes occur, it is safest to exhibit the maximum amount of corrosive and scale-forming ingredients which the water under these circumstances could develop. Thus, since calcium sulfate is practically insoluble in water above 100°, the proportion of calcium sulfate may be regarded as such as would be formed by the total quantity of calcium or the total quantity of SO_4, according to which is present in the larger ratio. Similarly, as the decomposition of magnesium chlorid is induced by the high temperature of the boiler, the analytic statement should indicate the maximum proportion of this compound obtainable from the magnesium and chlorin present. These rules

can not apply absolutely to waters rich in alkali carbonates, since these would neutralize any acid formed from the magnesium chlorid, or even prevent its formation, and would prevent, to a large extent, the formation of calcium sulfate. Much remains to be determined concerning the effects of the high temperature and concentration to which boiler waters are subjected.

General Uses.—In regard to the quality of water for other than steam-making purposes, such as brewing, dyeing, tanning, etc., no detailed methods or standards can be laid down. The nearest approach to purity that can be secured in the supply will be of the greatest advantage. The more objectionable qualities will be large proportion of organic matter, especially if it distinctly colors the water, excessive hardness, and notable amounts of iron or free mineral acid. It is stated that 1 part of iron per million will render water unsuitable for bleaching establishments. It has been noted that a large proportion of active microbes is injurious in the manufacture of indigo. In artificial ice making, a very pure water must be used if a clear and colorless product be desired. Any suspended or dissolved coloring-matter will be concentrated by the freezing and appear in the bottom or center of the mass.

BIOLOGIC EXAMINATIONS.

In a comprehensive sense the living organisms of water include representatives of all the great groups of animals and plants. The higher forms are absent from very foul water. From an analytic point of view, observation is limited to the determinations of those forms which are inappreciable to the unassisted eye. So far as regards some of the moderately complex organisms, such as the minute crustaceans, algæ, desmids, and even the amebæ, it may be said that while some general inferences as to the character and history of the water may be deduced from an identification of the specific forms, no definite sanitary signification can be attached to them. From what is now known of the life-history of many parasitic organisms, it is evident that water that is freely accessible to any animal forms is liable to be dangerously polluted. Moreover, the dead bodies of such animals will furnish food to many forms of microbes and thus assist in the multiplication of the latter. The ova of the entozoa might in some cases be detected by careful search, and would indicate recent pollution of a highly dangerous character.

The number of the higher forms present in any sample will depend very much upon the point

at which it is collected, they being more numerous in the neighborhood of large plants and at the bottom and sides of streams.

Several observers, notably Sedgwick and Rafter, have paid considerable attention to the recognition of the animal and vegetable forms in surface waters. Some of these forms cause disagreeable odors and colors; in the warm season of the year, when such water is stored in reservoirs, considerable annoyance is felt by the users, and the engineer-in-charge is subjected to much criticism. It has been found that even crude filtration methods, such as allowing the water to pass through a dike of porous soil before storing it in a reservoir, will diminish the tendency to these conditions. Cleansing a reservoir, disinfecting the inner surface, for instance, by whitewashing, has also improved the condition.

Observation, especially in Massachusetts, has shown that reservoirs intended for even moderately prolonged storage of water should be clean —that is, organic matter of any kind should not be allowed to accumulate on the bottom and sides. Drown states that while the water in one basin became foul from stagnation, in another which was carefully prepared by the removal of all soil and vegetable matter, and is supplied by a brown, swampy water from a

district almost entirely free from pollution, the water is good at a depth of 40 feet.

In Philadelphia, large storage reservoirs were used for water, often very muddy, but little trouble from the growth of microscopic organisms occurred. These reservoirs are artificial basins. They are now mostly used for filtered water.

Sedgwick's method of collecting organisms other than microbes, with some modifications by Williston, is as follows:

In ordinary cases about 100 c.c. are employed. Sometimes it will be advantageous to use double this quantity, at other times much less. In rare cases the examination can be made upon unfiltered water. Originally sand was employed for a filter material, but Williston finds that precipitated silica, made by decomposing silicon fluorid with water, is more satisfactory. This precipitated silica is a commercial article, and its method of preparation is given in all the larger manuals of chemistry.

A small glass funnel with an even-calibered stem is selected, and the lower end of the stem plugged with a little absorbent cotton, upon which a layer 4 mm. deep of the filter-material is placed. The water is passed, the pledget of cotton removed, and the filter-material washed down with filtered or distilled water into a cell intended for microscopic examination. This cell is a glass

plate accurately ruled, to which is attached a brass cell 50 mm. long by 10 mm. wide, of depth sufficient to hold about 2 c.c. of water. After the material has been allowed to distribute itself and settle in the cell, it is examined with a moderate power, and the different organisms in a

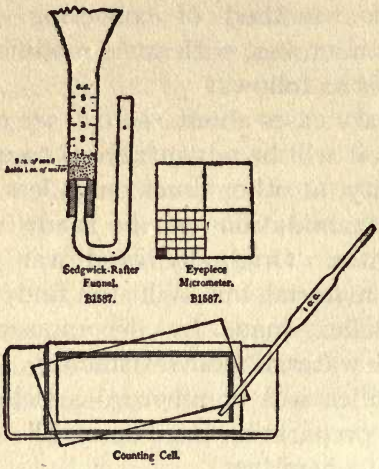

FIG. 6.

number of the squares counted. Each organism may be counted by itself, if occurring in large numbers, the average of a few squares being sufficient for the purpose. Organisms less numerously represented may be counted by averaging a larger number of squares. Fig. 6, cut loaned

by Arthur H. Thomas Co., shows a more elaborate form.

Filtering in this manner can not be relied upon in all cases. Indeed, in most cases the unfiltered water also should be examined. Some of the minute unicellular organisms pass readily through the small extent of sand or precipitated silica, or even through filter-paper.

It is not unlikely that the high-speed centrifugal apparatus now used in laboratories, associated with the employment of some fine precipitant, will aid in these investigations.

Owing to the great differences in the size of microscopic organisms, the mere enumeration of their numbers is not always an index of the amount of living matter in suspension. To obviate this, Whipple has suggested a standard unit of size, estimating by means of it the total volume of the organisms, and not their number. He finds by this method that the analytic and biologic results correspond much more closely than when mere numbers are recorded. The unit is an area of 400 microns—that is, a square of 20 microns one side. The results are stated in number of standard units per c.c.

Whipple has investigated the conditions influencing the growth of the microscopic organisms in water. He finds that diatoms thrive best with a supply of nitrates and a free circula-

tion of air; temperature alone has no very direct effect. Infusoria will be found in largest numbers when the water contains the greatest amount of finely divided organic matter. When the conditions bring about a circulation of the water, the organisms are not only brought constantly in contact with new food materials, but are enabled to reach the upper layers of the water where oxygen is abundant.

Bacteriologic examinations may be *qualitative* or *quantitative*. The former involves the determination of the species of microbes present, especially those having disease-producing power, or characteristic of some form of pollution. The processes are usually laborious, requiring extensive laboratory facilities. Quantitative examination—microbe-counting, as it may be called—is the determination of the number of microbes, or microbe-colonies, that can be grown from a given volume of water under specified conditions. As the growth of living organisms is influenced by all external conditions, results are not comparable, unless strict uniformity of method has been observed. Neglect of this fact renders a very large part of the earlier work and some of the present-day work of little statistical value. Among the conditions materially affecting the growth of microbes are temperature, reaction of the culture-medium to different indicators, de-

gree of exposure to light and air, and duration of cultivation. The composition of the culture-medium has much influence, and it is difficult to control this exactly, owing to the irregularity of quality of some of the materials used.

In the routine of water-analysis, bacteriologic examinations are chiefly determination of the number of living microbes in a given volume and detection of certain types. For the former procedure—microbe-counting—media solid at ordinary temperatures, but liquid at or about blood-heat, are employed, gelatin or agar being used. Gelatin-jelly melts below blood-heat (37°) but agar does not, hence, as will be seen by the formulas given below, the latter is almost entirely used. Apparatus and materials for bacteriologic work are now obtainable in standard form from dealers and only brief notice is needed.

The following constitute the equipment for water-bacteriology:

Open-steam sterilizer.

Autoclave.

Hot-air oven.

Incubating Oven with Thermostat.—Many forms are now in use, some heated by gas, some by electricity. The latter type has been brought to a high degree of efficiency in regulation of temperature, which is very important, as the oven

should not have a range of over 1° in operation at a given temperature.

Test-tubes.—About 12 cm. long and 1.5 cm. diameter.

Petri Dishes.—For the smaller sizes, earthenware covers are obtainable, which are better than glass, as they absorb the water given off from the culture, and which collecting in drops, may fall on the culture and produce confusion. In lack of earthenware covers, thick white blotting-paper fitted tightly to the inside of the glass will be of service.

In the early editions of this work, Leffmann and Beam described a method of "bottle-culture" in which flat rectangular bottles were used. The same idea is exemplified in "Kolle's flasks," flat glass flasks with a broad flat neck.

Ferment-tubes.—The standard dimensions are: closed arm about 15 cm. long, 1.5 cm. diameter; bulb about 4 cm. in diameter, it being intended that the bulb shall be able to hold all the liquid from the longer arm.

Wire baskets for holding test-tubes.

Ordinary laboratory apparatus, such as pipets, burets, funnels, beakers, cotton-wad, tinfoil cut in squares, 5 cm. on the side. A double-boiler, of which the inner vessel will hold about 2000 c.c., is needed.

Gelatin.—Some bacteriologists use a French

gelatin (Coignet) but most prefer a special form sometimes termed "gold-label gelatin" manufactured in Germany. Much uncertainty, however, prevails in regard to the source and quality of the gelatin sold to bacteriologists, and it will be safest to purchase from some trustworthy dealer who is in touch with the manufacturers. Good gelatins of American manufacture are obtainable, but bacteriologists do not use them. The term "gold label" has no value as any one may use such a label.

Gelatin and agar often contain notable amounts of moisture, and it is recommended that each should be assayed by drying a small, known, weight at 105°, and allowing for the moisture thus ascertained in weighing out portions for media.

Agar.—The ordinary article termed "thread agar," is satisfactory. Powdered agar is made in Germany; it is more expensive, but dissolves more quickly.

Peptone.—Witte's dry peptone is generally recommended, but a good peptone is made in the United States.

Glucose.—The grade termed "crystallized, pure;" it consists mostly of dextrose.

Meat-extract.—It is customary to recommend "Liebig's extract," but this term has no positive value as any one can so designate his product.

It is probably intended to limit the choice to the product of the Liebig Extract of Meat Co., of London, but there is no reason to give this preference over the American extracts.

Sodium Chlorid.—A good quality of table-salt will serve.

Lactose.—This should be of high purity, especially free from protein matters and other carbohydrates.

Preparation of Culture-media:

Broth (*Bouillon*).—Broths are fluid media, the basis of ordinary broth being the soluble portions of raw beef. Some operators prefer to obtain this by macerating meat in cold water for a number of hours, but the general custom is now to use commercial meat-extracts. A standard broth is made as follows:

Meat-extract.....	3 grams
Peptone.....	10 grams
Sodium chlorid.....	5 grams
Water.....	900 c.c.

The ingredients are heated in a double-boiler until dissolved, made up to 1000 c.c. and the reaction ascertained as described on page 99. The APHA deprecates the use of meat-extract, but nevertheless has substituted it for meat-infusion in several cases without giving any reason for such action.

Reaction of Culture-media.—Culture-media are now brought to a standard reaction, expressed in per cent. of $^{N}/_{1}$ acid or alkali required to make the solution neutral, acid media being marked +, and alkaline media −. The reaction used by American bacteriologists is + 1 per cent. that is, 100 c.c. of the media should require 1 c.c. of $^{N}/_{1}$ alkali to produce neutrality. Phenolphthalein (0.5 c.c. to 100 c.c. of 50 per cent. alcohol) must be used as the indicator.

Determination is as follows: 5 c.c. of the medium are mixed with 45 c.c. of water, the mixture boiled briskly for one minute, 1 c.c. of the indicator solution added and the liquid titrated, while boiling, with $^{N}/_{20}$ sodium hydroxid to a faint but distinct pink. The amount of alkali to be added to the whole volume of the medium to bring the acidity down to + 1 per cent. can be easily calculated.

If it is desired to use meat as the basis of broth, 500 grams of beef as free as possible from bone, gristle and fat, should be finely divided in a meat-chopper, and macerated overnight in about 500 c.c. of water between 0° and 10°. The liquid is strained through cloth, obtaining as much liquid as possible, and substituted for the meat extract, the amount of meat here given being adapted to make 1000 c.c. of medium. The mixture, before taking the reaction, must be

heated in the open-steam sterilizer to coagulate albumin, and then filtered.

Plain broth is but little used; it is generally enriched with special ingredients as given below. In many of these special forms the sodium chlorid is omitted.

Culture-media should be clear. Filtration, often difficult, is best carried out in a hot-water funnel. Filters of cotton-wool or cotton cloth may be used.

Fermentations.—Under this term is included the action of microbes producing transformation of carbohydrates by which notable amounts of gas and acids are produced. The gas is recognized and measured by performing the tests in ferment-tubes, which are filled with the nutritive medium, generally a sensitized broth.

The bulk of the gas is carbon dioxid and hydrogen, but other gases such as methane may be produced. Attempts to differentiate microbes by determining the ratio of the different gases have not been successful.

Ferment-tubes should be well cleaned, sterilized in open-steam, charged with the sterilized sensitized broth in such manner that the longer arm of the tube is quite filled, while the level of the liquid in the short arm is such as to permit the introduction of the material to be cultivated

and allow the overflow when the gas begins to collect in the long arm.

Fermentations are usually made at 37° for not less than forty-eight hours. They are often applied as a preliminary cultivation, inoculation being made from the ferment-tube into other media.

Solid Culture-media.—The elaborate methods on page 102 are given by the APHA. The meat should be finely divided in a meat-chopper. It is evident from the present-day literature that American bacteriologists are using meat-extracts largely in these solid media instead of meat-infusion, 3 grams of a good commercial extract being used for 1000 c.c. of medium. It is not clear what object is served by the several adjustments of reaction. It would seem that an adjustment when ready to sterilize will suffice.

Litmus-lactose-agar is prepared in the same manner as nutrient agar with the exception that 1 per cent. of lactose and a small amount of azolitmin are added just before sterilization. Kahlbaum's azolitmin is preferred, as it is readily soluble and does not alter the reaction.

Lactose-broth.—This contains 1 per cent. of lactose. A sufficient approximation will be obtained by adding 10 grams of lactose to 1000 c.c. of broth, prepared as described on page 98. It

is recommended that the mixture be made neutral to phenolphthalein.

	Gelatin.	Agar.
1.		Boil 15 grams agar in 500 c.c. of water for 30 minutes, or digest for 15 minutes in an autoclave. Make up the weight to 500 grams and allow the mixture to cool to 60°.
2.	Macerate 500 grams lean meat for 24 hours in 1000 c.c. of water at 0°.	Macerate 500 grams lean meat in 500 c.c. of water for 24 hours at 0°.
3.	Make up loss by evaporation.	
4.	Strain liquid through cotton cloth.	
5.	Weigh filtrate.	
6.	Add 1% of peptone and 10% of gelatin, both on dry basis.	Add 2% of peptone.
7.	Warm on water-bath (double-boiler) with stirring until substances are dissolved, at not over 60°.	
8.		To 500 grams of the meat solution add 500 c.c. of agar solution at not over 60°.
9.	Titrate after boiling for 1 minute.	
10.	Adjust reaction to + 1% by adding N/1 hydrochloric acid or sodium hydroxid as required.	
11.	Heat over boiling water for 40 minutes.	
12.	Restore loss by evaporation; readjust reaction to +1%, if necessary, boil 5 minutes over free flame.	
13.		
14.	Make up loss by evaporation.	
15.	Filter through absorbent cotton and cotton cloth, refiltering until clear.	
16.	Titrate and record final reaction.	
17.	Place in tubes (10 c.c. in each).	
18.	Sterilize 15 minutes in autoclave at 120° or in streaming steam for 30 minutes on three successive days.	
19.	Put tubes promptly in ice-chest in moist atmosphere.	Store in ice-chest in moist atmosphere.

Litmus-milk.—Fresh milk of the purest attainable quality is placed over night in the ice-chest, the cream removed as closely as possible, and the skimmed milk siphoned from any sediment. (It would seem that separator-skim could be substituted without detriment). The reaction must be + 1 per cent. 1 per cent. of azolitmin is added and the material promptly tubed and sterilized.

Lactose-bile.—Jackson, who has worked largely with this medium, gives data for its preparation in best form. Commercial inspissated bile is not very satisfactory. Fresh ox-bile filtered and sterilized in the autoclave for thirty minutes at 120° will keep for a long while. Bile also may be evaporated to dryness at a low temperature and preserved in this form. On an average 1000 c.c. of bile will yield 110 grams of residue, and therefore, 11 grams of it will suffice for 100 c.c. of solution. 1 gram of peptone and 1 gram of lactose are added to 100 c.c. of bile solution.

Hesse-agar.—

Agar (dried at 105° for 30 minutes),	4.5 grams
Peptone,	10.0 "
Meat-extract,	5.0 "
Sodium chlorid,	8.5 "
Water,	1000 c.c.

The agar is dissolved in 500 c.c. of the water; the other ingredients are dissolved in the remainder, the solutions mixed, boiled for thirty minutes, cooled, made up to 1000 c.c., and filtered through absorbent cotton held in the funnel by cotton cloth. The liquid should be clear. The reaction is adjusted to + 1 per cent. The medium is sterilized in tubes containing 10 c.c. each at 120° for twenty minutes, after which it should be cooled as quickly as possible and kept in a cool, moist atmosphere.

Endo's Medium.—This is enriched with decolorized rosanilin, under such conditions that the typhoid bacillus removes the decolorizing influence and the colonies appear bright-red. The APHA formula differs measurably from that given by Endo, being based on a formula advised by Kendall and Walker and used by them for isolating a dysentery microbe from intestinal discharges. The following formula is substantially that given by the APHA, except some unnecessary manipulations.

Agar,..... 30 grams (Endo used 40 grams)
Peptone,.....10 grams
Meat-extract, 5 grams (Endo used beef-infusion)

The materials are heated in a double-boiler with about 1000 c.c. of water until dissolved,

sodium carbonate added until the mass is neutral to litmus (azolitmin), 10 c.c. of a 10 per cent. solution of sodium carbonate added, the liquid made up to 1000 c.c., charged in portions of 100 c.c. into 150 c.c. flasks and heated for two hours in open-steam.

For enriching, the following solutions are used: Water 100 c.c., sodium sulfite, anhydrous, 1 gram. Alcohol (95 per cent.) 100 c.c., basic fuchsin, 1 gram.

The sulfite solution must be fresh; the fuchsin solution must be clear.

Add 2 c.c. of the fuchsin solution to 10 c.c. of the sulfite solution, steam the mixture for a few minutes, and add 0.5 c.c. of it 1 gram of lactose to each 100 c.c. of the unsensitized base, melt in open-steam, pour plates and allow to cool in the incubator. The solidified mass should be clear. Endo inoculated the medium by means of a glass rod with one end bent at a right angle, infecting this end with the material to be cultivated and smearing it over the surface of the medium.

Endo medium should not be enriched unless intended for prompt use; it should be kept in the dark.

Esculin Medium.—This is a special medium devised by Harrison and Leck for recognition of the typhoid bacillus.

Esculin,	0.1	gram
Peptone,	1.0	"
Sodium taurocholate,	0.1	"
Iron citrate,	0.05	"
Water,	100.0	c.c.

This mixture is sterilized, inoculated (see page 108) and plated in the usual way. It should not be made up until about to be used. The growth of the typhoid bacillus causes a reaction between the esculin and iron citrate by which black spots are produced at points at which the growth is taking place.

Indol Reaction.—Indol, C_8H_7N, more properly indin, is a weak base, which is a product of the growth of many species of microbes, and the detection of it may, therefore, be utilized as a differentiation test. Kitasato gives the following method for performing the test:

10 c.c. of plain broth which has been previously inoculated with the microbes to be tested, and kept for twenty-four hours at 37°, are treated with 1 c.c. of solution of pure potassium nitrite (0.02 gram in 100 c.c.) and then with a few drops of concentrated sulfuric acid. In the presence of indol a rose or deep red is developed. *Bacillus coli communis* gives the reaction strongly; *B. typhosus* ordinarily does not give it.

Culture Procedures.

Cultivation in gelatin at room temperature usually yields a larger number of points of microbic life than in agar at blood heat, due to the fact that many microbes do not grow well at the higher temperature.

Culture-media when ready for use are usually distributed in test-tubes. These must be well cleaned. In laboratories in which general chemical work is also carried on, the mixture of crude chromic and sulfuric acids used for voltaic batteries is convenient and efficient, the tubes being soaked in this for about a day and then thoroughly rinsed. Many bacteriologists use a weak solution of sodium hydroxid. The common lye sold for domestic soap-making is quite satisfactory. A couple of grams to 100 c.c. of tap-water will serve. The tubes are boiled in the solution, swabbed, rinsed and allowed to dry in an inverted position. A cotton plug is prepared for each tube care being taken that it contains no creases and does not fit too tightly. The projecting part of the plug is clipped moderately close and a tinfoil cap placed on each. The arrangement is sterilized in the hot-air oven at 150° C. When cold, the tinfoil and plug are carefully removed, 10 c.c. of culture-medium put into each tube, with as little outside contami-

nation as possible, the plug and cap replaced, and the tubes and contents sterilized in the open-steam sterilizer. Wire baskets are used to hold the tubes during the sterilizations.

For making cultures definite volumes of the water sample are introduced into the culture-medium, and if this is a solidifying form, it is put into the Petri dish. All the manipulations must be conducted with great care to avoid contamination. When there is no clue to the amount of microbes present, it will be necessary to make cultures with different proportions of water. Some tubes may be inoculated with a few drops, some with three to five drops, and some with 1 c.c. Some operators dilute the water considerably and take small measured volumes. If this method is used, the diluting water must be distilled and have been well sterilized, by at least five minutes' boiling, and cooled out of contact of air. All pipets, dishes, and other apparatus that come in contact with the water must have been first sterilized.

The test-tubes containing the culture-medium are warmed gently, just enough to render the culture-medium fluid, the desired volume of the water added, the mixture shaken and promptly poured into the Petri dishes, covered and placed in the oven, which should have been already raised to the temperature at which it is desired

to conduct the work. Unless specially desired otherwise, cultures should be made in the dark. They may be made at any temperature short of that at which the medium melts, but either ordinary temperature or 37° is usually selected. The condition of the plates should be observed at intervals of twenty-four hours, and the points of microbic life counted and recorded. After some days the growth will become so luxuriant or the liquefaction of the medium so extensive that accurate observation is not possible.

Cultivation in flat rectangular bottles or Kolle's flasks (page 96) involves simpler manipulations and less risk of contamination, especially with molds, which are serious accidental infections in this work. Sufficient of the medium is put into each bottle or flask to cover the flat surface to the depth of a few mm., the cotton plug is inserted and the vessel and contents sterilized as usual, that is, either for fifteen minutes in the autoclave at 120°, or for twenty minutes on three successive days in the open-steam apparatus. The medium is cooled to just above its melting point, the proper quantity of water put in, mixed well, the bottle or flask laid on the side to cool and then incubated at the proper temperature.

If the microbic points are numerous, it will be necessary to employ a counting scale. For the

Petri dish, Pake's modification of Lafar's scale is cheap and sufficient.

General Character of the Microbes in Natural Waters.—The microörganisms of natural waters are principally included in the genera *Bacillus* and *Spirillum*, especially the former. Micrococci and molds are rare, and are generally due to contamination by dust.

Microbes are also differentiated by the effects which they produce upon the culture-medium. Some species rapidly or slowly liquefy the jelly with evolution of foul-smelling gases; others—chromogenic microbes—produce characteristic colors. Many do not produce any positive modification, and for purposes of distinction it is usual to transfer colonies to other culture media. Such transfers are made by taking up a small portion of the culture on a loop of wire that has just been sterilized in the flame, and introducing this into the culture-medium.

Practical Application of Bacteriologic Methods.—As noted previously, the problems presented in the commercial or works-laboratory in relation to the bacteriology of water are the determination of the number of living organisms in a given volume of the sample, the identification and, if possible, isolation of specific disease-producing forms. Of the multitude of procedures devised for these purposes, a few only deserve special

mention. These are cultivation in nutrient agar (page 102) at 37°, for microbe counting, in broths enriched with carbohydrates to detect fermenting (gas-producing and acid-producing forms), cultivation in milk to detect coagulating forms, and in bile-broths for further differentiation, especially between the bacilli of the colon group and the true germ of typhoid fever.

The following routine for the examination of water supplied by corporations engaged in interstate commerce, has been recently issued by the U. S. Public Health Service, and may be regarded as a practical series of procedures. For interpretation of these results see page 127.

Cultivation in standard agar (page 102) for twenty-four hours at 37°, at least two plates being set.

Cultivation of portions of 10 c.c. in ferment-tubes in lactose-peptone-broth (page 101) at 37° for forty-eight hours, not less than five tubes being set.

Cultivation in litmus-lactose-agar (page 101) for twenty-four hours at 37°.

Inoculation of material from ferment-tubes into Endo medium (page 104) plates, and subsequent cultivation of typical colonies from these inoculations in ferment-tubes containing lactose-peptone-broth, and the percentage volume of gas noted.

Jackson has worked out a process for identification of the typhoid bacillus depending on the use of Hesse-agar.

The sample is cultivated for at least twenty-four hours in lactose bile (page 103). Eight tubes containing sterilized distilled water are arranged in a rack with eight Petri dishes, each set being designated by numbers from 1 to 8. In tube 1 is placed 1 c.c. of the culture, the contents of the tube are well mixed, 1 c.c. placed in dish 1, and 1 c.c. in tube 2. The contents of tube 2 are mixed, 1 c.c. put in dish 2 and 1 c.c. transferred to tube 3. The dilution is carried on in this manner until the series is completed. Each dish is then charged with 10 c.c. Hesse-agar (page 103) which has been melted and cooled to 40°, the content of each dish is well mixed, chilled until solid and incubated in a moist atmosphere at 37° for twenty-four hours.

By this method characteristic growths are obtained from the typhoid bacillus but only when the dilution is high enough to give but few colonies to the dish. It is distinguished from the bacilli of the colon group by forming colonies of much larger size, often serveral centimeters in diameter, showing a broad translucent, or scarcely turbid, zone between the white center and the narrow white border.

Colonies may be taken off for identification by

other cultures as noted on page 104 or for microscopic examination.

The following compositions between typical *B. coli communis* and *B. typhosus* are given by Abbott.

Characteristics.	B. Typhosus.	B. Coli Communis.
Motility,.....	Conspicuous.	Not marked.
Growth in gelatin,..	Slow.	Not very slow.
Growth in milk,....	No coagulation; no acidity.	Acidity and coagulations in 48 hours at 37°.
Growth in liquid media containing glucose, lactose, or sucrose,.....	No evolution of gas.	Marked evolution of gas.
Growth in solid media containing lactose and litmus,.....	Colonies pale blue; no reddening of surrounding medium.	Colonies pink; surrounding medium red.
Indol reaction in peptone solution (48 hours at 37°.)	Rarely present.	Always present.

INTERPRETATION OF RESULTS.

STATEMENT OF ANALYSIS.

The composition of water is generally expressed in terms of a unit of weight in a definite volume of liquid, but much difference exists as to the standard used. The decimal system is very largely employed, the proportions being expressed in milligrams per 1000 c.c., nominally parts per million; or in centigrams per liter, nominally parts per hundred thousand. The figures are often given in grains per imperial gallon of 70,000 grains, or the U. S. gallon of 58,328 grains. In this work the composition is always expressed in milligrams per 1000 c.c. This ratio is practically equivalent to parts per million, except in case of water very rich in solids, 1000 c.c. of which will weigh notably more than one million milligrams. Factors for converting the different ratios are given at the end of the book.

From the analysis of a water it is rarely possible to ascertain the exact arrangement of the elements determined, but it is the custom to assume arrangements based upon the rule of associating in combination elements having the highest af-

finities, modifying this system by any inferences derived from the character or reactions of the water itself. It is preferable to express the composition of a water by the proportion of each ion present. In this way a water containing K_2SO_4 will be expressed in terms of K and SO_4, respectively. In the case of bodies like CO_2 and SiO_2, which may possibly exist free in the water, their proportion is expressed as such. It frequently occurs that the characteristics of some of the compounds in a water are sufficiently marked to indicate their presence, and there can be no objection to suggesting, in connection with the analytic statement, the inferences which may thus be drawn.

The organic matters, or derived products, are best stated in terms of the nitrogen which they contain, thus permitting a comparison of the different stages of decomposition. It is inadvisable to represent the amount of unchanged organic matter in terms of oxalic acid, as has been suggested, or to express the nitrogen in terms of albumin, or any other supposititious compound.

The results of bacteriologic examinations should, as a rule, be reported as "points of microbic life" in the given volume of water. Many operators, however, report the number of points as "colonies" or even "bacteria."

SANITARY APPLICATIONS.

Judgment upon the analytic results from a given sample of water depends upon the class to which it belongs, and to the particular influences to which it has been subjected. A proportion of total solids which would be suspicious in a rain or river water, would be without significance in that from an artesian well. On the other hand, a subsoil water of unobjectionable character would contain a proportion of nitrates which would be inadmissible in the case of a river or deep water. Location has also much bearing in the case; subsoil waters near the sea will be found to contain, without invoking suspicion, proportions of chlorin which would be ample to condemn the same sample if derived from a point far inland. Hence the importance of recording, at the time of collection, all ascertainable information as to the surroundings and probable source of the water.

Analyses of surface waters have little value, unless supplemented by a careful survey of the watershed to determine sources of pollution. Such survey will often discover conditions sufficient to condemn the supply, even though the analyses may be satisfactory. Indeed, it may be taken as a fundamental principle that surface water from even a sparsely populated district will be unsafe for use unless efficiently filtered.

Color, Odor, and Taste.—Water of the highest purity will be clear, colorless, odorless, and nearly tasteless. While in some cases a decided departure from this standard may give rise to suspicion, analytic observations are necessary to decide the point. Water highly charged with mineral matters will possess decided taste, vegetable matters may communicate distinct color; but, on the other hand, it may be highly contaminated with dangerous substances and give no indications to the senses. Well-waters occasionally become offensive in odor, from penetration of tree roots. The odor often recalls that of hydrogen sulfid. Sulfids are, indeed, often formed in such cases by the abstraction of oxygen from sulfates under the influence of microbes. Such waters are often used without apparent injury, but it is probable that if direct pollution occurs, the danger would be enhanced by the presence of the vegetable matter.

Surface waters collected in reservoirs or ponds often become very offensive from the growth of algæ, but apart from the disgust created by the water, it is not known that any harmful results occur to those using it.

Turbidity may be due to several causes, of different degrees of danger, but is always objectionable.

Total Solids.—Excessive proportions of mineral solids, especially of marked physiologic action, are known to render water non-potable, but no absolute maximum or minimum can be assigned as the limit of safety. Distilled water and waters very highly charged with mineral matter have been used for long periods without ill effects. The popular notion that the so-called hard waters conduce to the formation of urinary calculi is not borne out by clinical experience or statistical inquiry. Many urinary calculi are composed of uric acid, and are the results of disorders of the general nutritive functions.

Sanitary authorities have fixed an arbitrary limit of total solids of about 600 parts per million, but many artesian waters in constant use exceed this. An instance is found in the well on Black's Island, near Philadelphia, which contains nearly 1200 parts per million, is very agreeable in taste, and has been in constant use for some years by a number of persons without injury. The assertion that water to be wholesome must contain an appreciable proportion of total solids is also not demonstrated by clinical experience. A discussion of the effects of special mineral ingredients—*e. g.*, magnesium sulfate, ferrous carbonate, etc.—belongs to general therapeutics.

The odor produced on heating the water is

often of much use in detecting contamination. Odors similar to those produced by heating glue, hair, rancid fats, urine, or other animal products, will give rise to grave suspicion. On the other hand, a more favorable judgment may be given when the odor recalls those given off in the heating of non-nitrogenous vegetable materials, such as wood-fiber.

Poisonous Metals.—The proportion of iron in water constantly used for drinking purposes should not much exceed 3 parts per million. Lead, copper, arsenic, and zinc must be considered dangerous in any amount, though it appears that zinc and copper, being least cumulative, are rather less objectionable in minute amount than the others. Concerning the limit of safety with manganese and chromium very little is known, but their presence in appreciable quantity must be looked upon with suspicion.

Chlorids and Phosphates.—Chlorids—principally sodium chlorid—and phosphates are abundantly distributed in rocks and soils, and find their way into natural waters; but while the former are freely soluble and remain in undiminished amount under all conditions to which the water is subjected, all but small amounts of the latter are either precipitated or removed by the action of living organisms. Surface and subsoil waters ordinarily contain but a few parts per

million. Both chlorids and phosphates being constant and characteristic ingredients of animal excretions, it is obvious that an excess of them in natural waters, unless otherwise accounted for, will suggest direct contamination. Proximity to localities in which sodium chlorid is abundant, such as the sea- or salt-deposits, will deprive the figure for the chlorin of diagnostic value, nor can any indication of sewage or other dangerous pollution be inferred from high proportion of chlorin in deep waters. Further, it has been shown that the proportion of chlorin in uncontaminated waters is tolerably constant, while in water subjected to the infiltration of sewage the chlorin undergoes marked variation in amount. In most cases, therefore, a correct judgment can only be attained by comparison with the average character of the waters of the same type in the district, and by examination at intervals of the water in question.

As regards phosphates, Hehner, who has published a series of analyses, states that the presence of more than 0.6 part per million—calculated as PO_4—should be regarded with suspicion. On the other hand, the absence of phosphates affords no positive proof of the freedom from pollution. Woodman, who has carefully investigated this question, regards Hehner's limit as too strict. He would fix 1 part per million as the minimum.

He regards this datum as valuable in judging of the sanitary quality of the sample.

Nitrogen from Ammonium Compounds ("Free Ammonia").—Ammonium compounds are usually the results of the putrefactive fermentation of nitrogenous organic matter; they may also be the product of the reduction of nitrites and nitrates in presence of excess of organic matter. In either case, therefore, they suggest contamination. Deep waters often contain an excess of ammonium compounds, derived, in large part, from the reduction of nitrates. Their presence here is hardly ground for adverse judgment, since the water, even though originally contaminated, has undergone extensive filtration and oxidation, its organic matter converted into bodies presumably harmless, and microbes have perished. Such waters, indeed, usually show only traces of unchanged organic matter.

Rain water often contains large proportions of ammonium compounds; but here, also, the fact can not condemn the water, since it does not indicate contamination with dangerous organic matter.

The evolution of ammonia in the distillation of rain water may continue indefinitely, the larger portion passing over in the first distillates, but small quantities being present even after the distillation has been much prolonged. The same

continuous evolution of ammonia has been noted in waters containing urea, but in this case a larger proportion is collected in the earlier distillates, nearly all coming over before one-half the water has been distilled.

Nitrogen by Alkaline Permanganate ("Albuminoid Ammonia").—A large yield of ammonia by boiling with alkaline potassium permanganate will, of course, point to an excess of nitrogenous organic matter. The inferences to be drawn depend upon the origin and condition of the organic material. If animal, the water may at once be condemned as unsafe. Waters containing excessive amounts even of vegetable matter are not free from objection, since they have frequently caused persistent diarrhea. If the organic matter, whether animal or vegetable, is in a state of active decomposition, it is doubly objectionable. Mallet has called attention to the fact that such waters, as a rule, yield ammonia rapidly, whereas non-decomposing material yields it but slowly, and he points out the importance, therefore, of noting the rate at which the ammonia collects in the distillate.

Smart has observed that water containing fermenting vegetable matter is colored yellow by boiling with sodium carbonate.

Inferences as to the source of the organic matter can usually be drawn from the amount of

chlorin and nitrates present. If the chlorin is high—*i. e.*, in excess of the average of the district—it may be inferred that the material is, in great part, of animal origin. In this case the nitrates will either be high or entirely absent, according as the contaminating matter has passed through soil or enters the water directly.

A large amount of vegetable matter will, as a rule, show itself by the color it imparts to the water.

Total Nitrogen.—Drown and Martin's results with surface waters indicate that the total nitrogen obtained by their process is about twice that obtained by alkaline permanganate. The experiments made by Leffmann and Beam accord with this. Further observation on different waters and by different observers will be required to determine the value to be assigned to the figures obtained by this method. This method is especially suitable for studying the effects of filtration, storage, etc., on the nitrogenous organic matter in water.

Nitrogen as Nitrites.—Nitrites are present in water as the result either of incomplete nitrification of ammonium, or the reduction of already formed nitrates, under the influence of reducing agents or microbes. Since they are transition products, their presence in water is usually evidence of existing fermentative changes,

and, further, may be taken as indicating that the water is unable to dispose of the organic contamination. When, however, the conditions are such that oxidation can not take place, nitrites may persist for a long time. This sometimes occurs in deep waters in which fermentative changes have long since ceased, but oxygen is not available. These contain not infrequently small amounts of nitrites, to which the same degree of suspicion can not be attached. When nitrites are found in these waters, the possibility of their introduction from polluted subsoil water, through defective tubing, must not be overlooked. Rain water, also, sometimes contains nitrites derived from the air, and therefore not indicative of any putrefactive change. The presence of measurable quantities of nitrites in river or subsoil water is sufficient ground for condemnation.

Nitrogen as Nitrates.—Nitrates are the final point in the oxidation of nitrogenous organic matter, especially animal matters. Rain water and that from mountain streams and deep wells, except from cretaceous strata, generally contains only traces, but river and subsoil waters will always contain appreciable amounts, unless some reducing action, such as recent sewage pollution, is at work. When, therefore, a water contains enough mineral matter to demonstrate its percolation through soil, and at the same time

is free from nitrates or contains only traces, the occurrence of a destructive fermentation may be inferred. These cases are not uncommon among well-waters, and the samples are generally turbid from suspended organic matter. Decided departure, either by increase or decrease, from the proportion of nitrates usual in the same class of water in any district may be taken as evidence of contamination.

Oxygen-consuming Power.—Sanitary authorities differ very much as to the significance of this datum. Attempts have been made to fix maximum limits for the various types of water, and also to gage the character and condition of the organic matter by observing the rate at which the oxidation takes place, but no positive conclusions can be given. In general, it may be said that a sample which has high oxygen-consuming power will be more likely to be unwholesome than one which is low in this respect; but the interferences are so numerous, and the susceptibility to oxidation of different organic matters, of even the same type, is so different, that the method is at best only of accessory value. It is especially suitable for consecutive determinations on the same supply.

For the method with acidified permanganate at the boiling heat, the German chemists, who employ it largely, regard an absorption of 2.5

parts of oxygen per million as suspicious, and some sanitary authorities have fixed 3.8 parts of oxygen per million as the highest permissible limit.

Dissolved Oxygen.—Full aëration of water is favorable to the destruction of organic matter; a decided diminution in the quantity of dissolved oxygen may show excess of such matter and of microbic life. Gérardin has pointed out that this diminution is associated with the development of low forms of vegetable life, and Leeds has recorded similar facts. These changes are more likely to take place in still waters, and are frequently accompanied by disagreeable odor and taste. In cases in which stored waters become unpalatable, these facts should be borne in mind.

Hardness.—The degree of hardness, unless very high, has but little bearing on the sanitary value of water, but is important in reference to its use for general household purposes, in view of the soap-destroying power which hard waters possess.

USUAL ANALYTIC RESULTS FROM UNCONTAMINATED WATERS.

Milligrams per Liter.

	Rain.	Surface.	Subsoil.	Deep.
Total solids,	5 to 20	15 upward	30 upward	45 upward
Chlorin,	Traces to 1	1 to 10	2 to 12	Traces to large quantity
Nitrogen by permanganate.	0.08 to 0.20	0.05 to 0.15	0.05 to 0.10	0.03 to 0.10
Nitrogen as NH_4	0.20 to 0.50	0.00 to 0.03	0.00 to 0.03	Generally high
Nitrogen as nitrites.	None or traces	None	None	None or traces
Nitrogen as nitrates.	Traces	0.75 to 1.25	1.5 to 5	0.00 to 3

Inferences from Cultures.—It is obviously impossible to fix a rational limit to the number of microbes (points of microbic life) permissible in a given sample, since season, temperature, extent of exposure to air and the time during which the sample has remained at a given temperature affect the development materially. Below a few meters from the surface, the soil is generally almost sterile, hence subsoil waters deriving their supply from highly polluted sources may show but few microbes. Deep-well water is generally sterile. Rain water is usually rich in microbes, surface water extremely variable.

In addition, media made with great care by different persons and with materials from different sources will show differences with the same sample. Many bacteriologists have fixed a limit of 100 living organisms to 1 c.c. This is obviously largely the influence of the number itself, round and somewhat imposing, but it has been made the standard by the U. S. Public Health Service which, in controlling water-supplies coming within the interstate powers of the Federal Government, has ruled that waters yielding more than 100 colonies per c.c. in standard agar at 37° for twenty-four hours (see page 102) are not safe; that not more than one in five tubes incubating 10 c.c. of the sample in lactose-peptone-broth shall show appreciable gas

in forty-eight hours; that cultivation in litmus-lactose-agar or Endo's medium for twenty-four hours at 37° shall not show red colonies. Further, when typical colonies from these agar-cultures are re-inoculated in lactose-peptone-broth, not more than 10 per cent. of the tube shall be occupied with gas after forty-eight hours at 37°.

Culture methods have special value in the examination of subsoil waters, and in determining the efficiency of systems of purification, being, far as the latter purpose is concerned, indispensable.

PURIFICATION OF WATER.

Drinking Water.—The most obvious method of purifying water is by distillation, but the process is too expensive for general use. It has been used for supplying vessels at sea and in tropic localities in which the natural waters may be contaminated with malarial or other germs. The majority of microbes are killed by short exposure to a temperature of 100°; hence, water may be purified, on a small scale, by simple boiling. Freezing does not have as beneficial an effect, many microbes retaining vitality for a long time in ice, and even at very low temperatures. For household purposes filters are in use, the most efficient being those in which the water passes

through the pores of unglazed porcelain. Even these, however, should be frequently cleaned or they will deliver a contaminated filtrate.

The purification of water on the large scale for either household or industrial purposes is an engineering problem, the details of which are not within the scope of this work. Several types of methods have been devised. One type depends on adding to the water substances that produce colloidal precipitates, that entangle the suspended matters, living and non-living, and, by making them more bulky, permit of removal by rapid filtration under pressure. Aluminum compounds, *e. g.*, aluminum sulfate, are mostly used. In most cases sufficient carbonates are present in the water to react, producing aluminum hydroxid, a colloidal mass that has attraction for many of the suspended matters, and even for some of the dissolved matters. If the water is low in carbonates, small amounts of lime may be added and the result will be obtained.

Filtration through porous materials, generally fine sand, at a slow rate permits of a biologic action—the growth of microbes in the upper layers of the sand by which the food of the microbes is exhausted and they perish. This is the preferred method for large-scale supplies.

It has been found advantageous in many cases to supplement the action of the filter by in-

troducing small amounts of germicides into the water before delivery to the distributing system. Many substances have been tried. One of the most efficient is ozone, but it is costly to prepare and difficult to mix with the water. At present the preferred substance is chlorin, introduced either in free state or in the form of the so-called "chlorid of lime," generally termed by chemists calcium hypochlorite, but even this name is not unchallenged. The article is cheap and of fairly constant quantity, the best commercial samples being capable of yielding about one-third their weight of chlorin. Very small amounts of chlorin suffice to kill all ordinary water microbes, and the use of this agent is now much in favor.

Purification of *boiler waters* is directed principally to diminishing corrosion and preventing scale. The former problem is discussed on pages 84, 85; the latter relates principally to the removal of the calcium carbonate and sulfate, magnesium carbonate and chlorid. Both carbonates are appreciably soluble in pure water. The ratio of 15 parts of calcium carbonate per 1,000,000 is usually stated to be the solubility, but Allen found that solutions can be obtained containing twice this amount. If the water contains carbonic acid, it will take up a much greater proportion of the carbonates, but in

this case they will be deposited from the solution by boiling. This has been accounted for by supposing the existence of soluble bicarbonates, which are decomposed by the boiling. Nearly all of these carbonates can be thrown out of solution by any means that will deprive the water of the carbonic acid. Sodium hydroxid is often employed for the purpose, and should be added in quantity just sufficient to form normal sodium carbonate. If there are present in the water calcium and magnesium chlorids and sulfates, these also will be decomposed and precipitated by the sodium carbonate so formed. If the amount of sodium carbonate formed is not sufficient to decompose all of these bodies, a sufficient quantity should be added with tbe sodium hydroxid to effect the complete decomposition. The precipitate is allowed to settle or filtered off.

In cases in which the feed-water is heated before it enters the boiler, it may only be necessary to add to the water sodium carbonate in quantity sufficient to decompose the calcium and magnesium chlorids and sulfates, since the heat alone will suffice to throw down the carbonates.

Care should be taken in these precipitations that no more sodium hydroxid is added than is required for the precipitation, since excess produces corrosion.

Soluble phosphates added to a water precipitate completely in a flocculent condition any calcium, magnesium, iron, or aluminum. This reaction can be best applied by using the commercial trisodium phosphate. By reason of the facility with which this substance loses a portion of its sodium to acids, it acts not only as a precipitant to the above materials, but will neutralize any free mineral acid present in the water. From evidence submitted by those who have used the process on the large scale, it appears that not only is no hard scale formed, but that scale already existing prior to its use is gradually disintegrated and removed with the sludge. Experiments indicate that no injury results from an excess of the material; but the economical employment of the method, especially with very hard waters, can only be based upon a correct analysis, and an estimation of the phosphate required for the precipitation. In many cases the composition of the water will be such that a partial precipitation will be sufficient.

Considerable success has been obtained by the use of fluorids as precipitants of scale-forming elements. Sodium aluminate has also been recommended. A preparation termed "permutite" made by fusing together kaolin, quartz and sodium carbonate is being much used.

Waters rich in ferrous compounds may be

purified by thorough aëration and filtration, the iron precipitating as ferric hydroxid.

IDENTIFICATION OF THE SOURCE OF WATER.

The determination of the course of underground streams, and of communications between collections of water, is often an important practical problem. In geologic and sanitary surveys, valuable information may occasionally be gained. The method generally pursued when connection between water at accessible points is to be detected, is to introduce at one point some substance not naturally existing in the water, and capable of recognition in small amount. Lithium compounds are among the best for this purpose. They are not frequent ingredients of natural waters, and are easily recognized by the spectroscope. Lithium chlorid is the most suitable. The quantity to be employed will vary with circumstances. It scarcely needs to be stated that the waters under examination should be carefully tested for lithium before using the method.

When the lithium method is inadmissible, recourse must be had to other substances of distinct character, such as strontium chlorid, but this possesses the disadvantage that a considerable amount may be rendered insoluble,

and thus lost in the ordinary transit through soil. Use has been made of organic coloring matters of high tinctorial power, one of the most suitable of which is fluorescein. This will communicate a characteristic and intense fluorescence to many thousand times its weight of water. The coloration is distinct only in alkaline liquids. Other colors, such as anilin-red, may be employed.

Vandenburgh made successful use of both methods in a suit growing out of use of a creek for the supply of Syracuse, N. Y. It was shown that the creek supplied a spring which was used by a manufacturing establishment.

A more important feature of the problem from a sanitary point of view is the determination of the source of a given current or collection of water, when such source is inaccessible. Problems of this character are not infrequent in large cities in which the systems of water-supply and drainage are defective, thus giving occasion to accumulations of water in cellars and similar places. Often, in these cases, no extended explorations can be made, by reason of the adjacent buildings and conflicting property interests, and the question may arise whether the water proceeds from a leaky hydrant, drain, sewer, or subsoil current. It is obvious that in the case of the collection of water in a cellar from causes other than surface washings or entrance of rain,

it must have passed through some distance of soil, and in built-up districts will almost certainly be charged with organic refuse. To correctly interpret the results, it will be necessary to know the general character of the subsoil water of the district and the composition of the public supply. As a rule, the transmission of water through moderate distances of soil will not materially increase the mineral constituents. Hence, if the sample contains an excess of dissolved matters as compared with the water-supply of the district, it may reasonably be inferred that it is derived from a drain, sewer, or subsoil current. In these investigations it will generally be sufficient to determine the total solids, odor on heating, chlorin, nitrates, and nitrites.

Occasionally, the analytic results will be ambiguous, and it is advisable to make examinations of more than one sample, since accidental circumstances, rain-fall, etc., may affect the composition of the water.

Instances of the contamination of water by unusual substances are occasionally noted, and these sometimes afford a clue to the source of the water. Among the instances of this kind within the experience of Dr. Beam and myself, may be noted the contamination with petroleum and with soap. In the former case it was evi-

dent that the contamination was from a leaky pipe connecting two refineries. In the latter it was shown to be derived from an adjoining building used as a laundry.

FACTORS FOR CALCULATION.

Parts per 100,000	× 0.7	=	Grains per Imperial gallon
Parts per 1,000,000	× 0.07	=	Grains per Imperial gallon
Parts per 100,000	× 0.583	=	Grains per U. S. gallon
Parts per 1,000,000	× 0.058	=	Grains per U. S. gallon
Grains per Imp. gal.	÷ 0.7	=	Parts per 100,000
Grains per Imp. gal.	÷ 0.07	=	Parts per 1,000,000
Grains per U. S. gal.	÷ 0.583	=	Parts per 100,000
Grains per U.S. gal.	÷ 0.058	=	Parts per 1,000,000

Al_2O_3	× 0.53	=	Al
AgCl	× 0.247	=	Cl
$BaSO_4$	× 0.588	=	Ba
$BaSO_4$	× 0.411	=	SO_4
$BaSO_4$	× 0.342	=	SO_3
CaO	× 0.715	=	Ca
CaO	× 1.78	=	$CaCO_3$
$CaCO_3$	× 0.4	=	Ca
Cl	× 1.64	=	NaCl
Fe_2O_3	× 0.7	=	Fe
KCl	× 0.525	=	K
K_2PtCl_6	× 0.16	=	K
K_2PtCl_6	× 0.306	=	KCl
$Mg_2P_2O_7$	× 0.218	=	Mg
$Mg_2P_2O_7$	× 0.854	=	PO_4
$Mg_2P_2O_7$	× 0.757	=	$MgCO_3$
MnS	× 0.633	=	Mn
NaCl	× 0.394	=	Na
N	× 4.42	=	NO_3
N	× 3.27	=	NO_2
N	× 1.22	=	NH_3
NH_3	× 0.823	=	N

ATOMIC WEIGHTS.

Expressed to nearest first decimal.

Aluminum	27.1	Magnesium	24.3
Barium	137.4	Manganese	54.9
Calcium	40.1	Nitrogen	14.0
Carbon	12.0	Oxygen	16.0
Chlorin[1]	35.5	Phosphorus	31.0
Chromium	52.0	Platinum	195.2
Copper	63.6	Potassium	39.1
Hydrogen[1]	1.0	Silver	107.9
Iodin	126.9	Sodium	23.1
Iron	55.9	Sulfur	32.0
Lead	207.1	Zinc	65.4
Lithium	7.0		

[1] More accurately, 35.46 and 1.008, respectively.

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